10085368.1 Page 2

FILE 'HOME' ENTERED AT 12:38:38 ON 24 SEP 2003

Uploading

THIS COMMAND NOT AVAILABLE IN THE CURRENT FILE Do you want to switch to the Registry File? Choice (Y/n):

Switching to the Registry File ...

Some commands only work in certain files. For example, the EXPAND command can only be used to look at the index in a file which has an index. Enter "HELP COMMANDS" at an arrow prompt (=>) for a list of commands which can be used in this file.

=> FILE REGISTRY

COST IN U.S. DOLLARS

SINCE FILE TOTAL ENTRY SESSION 0.42 0.42

FULL ESTIMATED COST

FILE 'REGISTRY' ENTERED AT 12:39:29 ON 24 SEP 2003 USE IS SUBJECT TO THE TERMS OF YOUR STN CUSTOMER AGREEMENT. PLEASE SEE "HELP USAGETERMS" FOR DETAILS. COPYRIGHT (C) 2003 American Chemical Society (ACS)

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STRUCTURE FILE UPDATES: 23 SEP 2003 HIGHEST RN 591719-82-3 DICTIONARY FILE UPDATES: 23 SEP 2003 HIGHEST RN 591719-82-3

TSCA INFORMATION NOW CURRENT THROUGH JULY 14, 2003

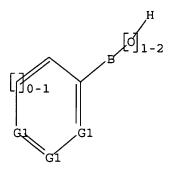
Please note that search-term pricing does apply when conducting SmartSELECT searches.

Crossover limits have been increased. See HELP CROSSOVER for details.

Experimental and calculated property data are now available. See HELP PROPERTIES for more information. See STNote 27, Searching Properties in the CAS Registry File, for complete details: http://www.cas.org/ONLINE/STN/STNOTES/stnotes27.pdf

Uploading 10085368.1

L1 STRUCTURE UPLOADED



G1 C,O,S,N,CH,NH

Structure attributes must be viewed using STN Express query preparation.

=> s 11

SAMPLE SEARCH INITIATED 12:39:45 FILE 'REGISTRY' SAMPLE SCREEN SEARCH COMPLETED - 1007 TO ITERATE

1000 ITERATIONS 99.3% PROCESSED

50 ANSWERS

INCOMPLETE SEARCH (SYSTEM LIMIT EXCEEDED)

SEARCH TIME: 00.00.01

FULL FILE PROJECTIONS: ONLINE **COMPLETE** BATCH

COMPLETE

PROJECTED ITERATIONS: 18237 TO 22043 6074

PROJECTED ANSWERS: 4156 TO

L2 50 SEA SSS SAM L1

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FULL SEARCH INITIATED 12:39:55 FILE 'REGISTRY' FULL SCREEN SEARCH COMPLETED - 19899 TO ITERATE

100.0% PROCESSED 19899 ITERATIONS 5071 ANSWERS

SEARCH TIME: 00.00.02

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=> file caplus

COST IN U.S. DOLLARS SINCE FILE TOTAL

ENTRY SESSION

FULL ESTIMATED COST 148.15 148.57

FILE 'CAPLUS' ENTERED AT 12:40:04 ON 24 SEP 2003 USE IS SUBJECT TO THE TERMS OF YOUR STN CUSTOMER AGREEMENT. PLEASE SEE "HELP USAGETERMS" FOR DETAILS. COPYRIGHT (C) 2003 AMERICAN CHEMICAL SOCIETY (ACS)

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FILE COVERS 1907 - 24 Sep 2003 VOL 139 ISS 13 FILE LAST UPDATED: 23 Sep 2003 (20030923/ED)

This file contains CAS Registry Numbers for easy and accurate substance identification.

=> s 13

L4 8602 L3

=> s 14 and benzene

L5 734 L4 AND BENZENE

=> s 14 and toulene

L6 0 L4 AND TOULENE

=> s 14 and phenyl

L7 2038 L4 AND PHENYL

=> s 15 and 17

L8 308 L5 AND L7

=> s l4 and pyridine

L9 1081 L4 AND PYRIDINE

=> s 14 and pyrimidine

L10 332 L4 AND PYRIMIDINE

=> s 14 and pyridazine

L11 81 L4 AND PYRIDAZINE

=> s 18 and 19 and 110 and 111

L12 5 L8 AND L9 AND L10 AND L11

=> d l12 fbib hitstr abs total

L12 ANSWER 1 OF 5 CAPLUS COPYRIGHT 2003 ACS on STN

AN 2002:676015 CAPLUS

DN 137:201315

TI Heteropolycyclic compounds, particularly pyridyl- and **phenyl**-substituted 1,2,4-oxadiazoles and analogs, and their use as metabotropic glutamate receptor antagonists for inhibiting neuronal damage

IN Slassi, Abdelmalik; Van Wagenen, Bradford; Stormann, Thomas M.; Moe, Scott
T.; Sheehan, Susan M.; McLeod, Donald A.; Smith, Daryl L.; Isaac, Methvin
Benjamin

PA Can.

SO PCT Int. Appl., 272 pp.

CODEN: PIXXD2

DT Patent

LA English

FAN.CNT 3

PATENT NO. KIND DATE APPLICATION NO. DATE

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     3-Fluorophenylboronic acid 1692-15-5, 4-Pyridylboronic acid
     10365-98-7, 3-Methoxyphenylboronic acid 30418-59-8,
     3-Aminophenylboronic acid 150255-96-2, 3-Cyanophenylboronic acid
     RL: RCT (Reactant); RACT (Reactant or reagent)
        (precursor; prepn. of pyridyl- and phenyl-substituted
        oxadiazoles and analogs as metabotropic glutamate receptor antagonists
        for inhibiting neuronal damage)
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RN
CN
     Boronic acid, phenyl- (9CI) (CA INDEX NAME)
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RN 768-35-4 CAPLUS CN Boronic acid, (3-fluorophenyl)- (9CI) (CA INDEX NAME)

RN 1692-15-5 CAPLUS CN Boronic acid, 4-pyridinyl- (9CI) (CA INDEX NAME)

RN 10365-98-7 CAPLUS CN Boronic acid, (3-methoxyphenyl)- (9CI) (CA INDEX NAME)

RN 30418-59-8 CAPLUS

CN Boronic acid, (3-aminophenyl) - (9CI) (CA INDEX NAME)

RN 150255-96-2 CAPLUS

CN Boronic acid, (3-cyanophenyl) - (9CI) (CA INDEX NAME)

GI

AB The invention provides compds. and pharmaceutical compns. that act as antagonists at metabotropic glutamate receptors, and that are useful for treating neurol. diseases and disorders. Methods of prepg. the compds. also are disclosed. The compds. exhibit a high degree of potency and selectivity for individual metabotropic glutamate receptor subtypes, notably mGluR5. In particular, medical conditions assocd. with metabotropic glutamate receptors and therefore targeted by the invention compds. include stroke, head trauma, anoxic injury, ischemic injury, hypoglycemia, epilepsy, pain, migraine headaches, Parkinson's disease, senile dementia, Huntington's Chorea, and Alzheimer's disease. The invention provides methods of treating diseases assocd. with excitatory activation of an mGluR Group I receptor, and of inhibiting neuronal damage

caused by excitatory activation of an mGluR Group I receptor, specifically wherein the mGluR Group I receptor is mGluR5. In one aspect of the invention, the antagonists may be represented by the general formula Ar1-L-Ar2, wherein Ar1 is an optionally substituted heteroarom. moiety, and Ar2 is an optionally substituted benzene ring. The L moiety is a group that not only covalently binds to the Arl and Ar2 moieties, and which facilitates adoption of the correct spatial orientation of Ar1 and Ar2, but also itself may interact with the protein, to effect receptor binding. In one embodiment of the invention, L is selected from the group consisting of -NH-, -S-, -O-, -CO-, -CONH-, -CONHCH2-, -CH2CONH-, -CNHNH-, -CNHNHCH2-, -C=NOCH2-, -CH2NHCH2-, -CH2CH2NH-, -NHCH2CO-, -NHCH2CHOH-, -NHCNHNH-, -NHCONH-, cyclopentane, cyclopentadiene, furan, thiofuran, pyrrolidine, pyrrole, 2-imidazoline, 3-imidazoline, 4-imidazoline, imidazole, pyrazoline, pyrazolidine, imidazolidine, oxazole, 2-oxazole, thiazole, isoxazole, isothiazole, 1H-1,2,4-triazole, 1H-1,2,3-triazole, 1,2,4-oxathiazole, 1,3,4-oxathiazole, 1,4,2-dioxazole, 1,4,2-oxathiazole, 1,2,4-oxadiazole, 1,2,4-thiadiazole, 1,2,5-oxadiazole, 1,2,5-thiadiazole, 1,3,4-oxadiazole, 1,3,4-thiadiazole, 1H-tetrazole, cyclohexane, piperidine, tetrahydropyridine, 1,4-dihydropyridine, pyridine, benzene, tetrahydropyran, 3,4-dihydro-2H-pyran, 2H-pyran, 4H-pyran, tetrahydrothiopyran, 3,4-dihydro-2H-thiopyran, 2H-thiin, 4H-thiopyran, morpholine, thiomorpholine, piperazine, pyridazine, pyrimidine, pyrazine, 1,2,4-triazine, 1,2,3-triazine, 1,3,5-triazine, and 1,2,4,5-tetrazine. In another embodiment of the invention, Arl is selected from the group consisting of Ph, benzyl, naphthyl, fluorenyl, anthrenyl, indenyl, phenanthrenyl, and benzonaphthenyl, and Ar2 is selected from the group consisting of thiazoyl, furyl, pyranyl, 2H-pyrrolyl, thienyl, pyrroyl, imidazoyl, pyrazoyl, pyridyl, pyrazinyl, pyrimidinyl, pyridazinyl, benzothiazole, benzimidazole, 3H-indolyl, indolyl, indazoyl, purinyl, quinolizinyl, isoquinolyl, quinolyl, phthalizinyl, naphthyridinyl, quinazolinyl, cinnolinyl, isothiazolyl, quinoxalinyl, indolizinyl, isoindolyl, benzothienyl, benzofuranyl, isobenzofuranyl, and chromenyl. Several hundred specific examples are individually prepd. and/or claimed. A variety of intermediates were also prepd. For instance, 5-methylpyrid-2-ylamidoxime was prepd. from 2-bromo-5-methylpyridine by Zn- and Pd-complex-mediated cyanation (56%) and reaction of the resulting nitrile with NH2OH.HCl (60%). Cyclization of the amidoxime with 3-cyanobenzoyl chloride (86%) gave invention compd. I. In a bioassay for mGluR5 antagonism in primary astrocyte cultures from rats, the invention compds. had IC50 values in th range of 11 to 9140 nM.

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L12 ANSWER 2 OF 5 CAPLUS COPYRIGHT 2003 ACS on STN
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- PA Eisai Co., Ltd., Japan
- SO PCT Int. Appl., 174 pp.

CODEN: PIXXD2

- DT Patent
- LA Japanese

FAN.CNT 1

PATENT NO. KIND DATE APPLICATION NO. DATE

AN 2002:220564 CAPLUS

DN 136:263177

TI Preparation of pyridazinones and triazinones exhibiting excellent inhibitory activities against AMPA receptor and/or kainate receptor

IN Nagato, Satoshi; Kawano, Koki; Ito, Koichi; Norimine, Yoshihiko; Ueno, Kohshi; Hanada, Takahisa; Amino, Hiroyuki; Ogo, Makoto; Hatakeyama, Shinji; Ueno, Masataka; Groom, Anthony John; Rivers, Leanne; Smith, Terence

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                        A1 20020321
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OS
     MARPAT 136:263177
     98-80-6, Phenylboronic acid 5720-06-9,
IT
     2-Methoxyphenylboronic acid 17933-03-8, m-Tolylboronic acid
     RL: RCT (Reactant); RACT (Reactant or reagent)
         (prepn. of pyridazinones and triazinones exhibiting excellent
         inhibitory activities against AMPA receptor and/or kainate receptor for
         treatment or prevention of acute or chronic neurodegenerative diseases)
RN
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     Boronic acid, phenyl- (9CI) (CA INDEX NAME)
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HO-B-OH
     5720-06-9 CAPLUS
RN
CN
     Boronic acid, (2-methoxyphenyl) - (9CI) (CA INDEX NAME)
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RN 17933-03-8 CAPLUS CN Boronic acid, (3-methylphenyl)- (9CI) (CA INDEX NAME)

GI

Ι

AB The title compds. [I; wherein Al, A2 and A3 are each independently C3-8 cycloalkyl, C3-8 cycloalkenyl, a 5- to 14-membered nonarom. heterocyclic group, a C6-14 arom. carbocyclic group, or a 5- to 14-membered arom. heterocyclic group, any of which may be substituted; Q is O, S, or NH; Z is C or N; X1, X2 and X3 are each independently a single bond, optionally substituted C1-6 alkylene, optionally substituted C2-6 alkenylene, optionally substituted C2-6 alkynylene, NH, O, NHCO, CONH, SO0-2, or the like; R1 and R2 are each independently hydrogen or optionally substituted C1-6 alkyl, or alternatively R1 and R2 may be united in such a way that CR2-ZR1 forms C:C; and R3 is hydrogen, optionally substituted C1-6 alkyl, C2-6 alkenyl, or C2-6 alkynyl, or alternatively R3 may unite with any atom on the ring A1 or A3 to form together with the atom an optionally substituted C5-8 carbocycle or an optionally substituted 5- to 8-membered heterocycle] or salts thereof, or hydrates of both are prepd. These compds. do not inhibit N-methyl-D-aspartic acid (NMDA) receptor but they are excellent inhibitors of .alpha.-amino-3-hydroxy-5-methyl-4isoxazolepropionic acid (AMPA) receptor and/or kainic acid receptor. are useful for the prevention or treatment of acute neurodegenerative diseases, acute cerebral vascular disorders, head injury, spinal cord injury, nerve disorders caused by low oxygen or sugar level, chronic neurodegenerative diseases, Alzheimer's disease, Parkinson's diseases, Huntington's chorea, amyotrophic lateral sclerosis, spinocerebellar

degeneration, epilepsy, hepatic encephalopathy, peripheral nerve disorder, Parkinson's syndrome, spastic hemiplegia (paralysis), pain, neuralgia, schizophrenia, anxiety, drug dependence, nausea, vomiting, urination disorder, eye sight disorder caused by glaucoma, hearing disorders caused by antibiotics, food poisoning, infectious encephalomyelitis (including HIV encephalomyelitis), cerebral vascular dementia, dementia caused by meningitis, and nerve diseases. They are also used for treatment or prevention of demyelinating diseases including encephalitis, acute disseminated encephalomyelitis, multiple sclerosis, acute multiple neuritis, Guillain-Barre syndrome, chronic inflammatory demyelinating multiple nerve disorders, Marchifava-Bignami disease, central bulbopontine breakdown, optic nerve myelitis, Devic's disease (neuromyelitis optica), Balo's disease, HIV myelopathy, HTLV myelopathy, progressive white substance encephalopathy or secondary demyelinating diseases (including central nervous system erythematodes, tuberous multiple polyarteritis, Sjoegren syndrome, sarcoidosis, or cerebral angiitis). Thus, to a soln. of 75 mg 2-(2-iodophenyl)-4-(3-pyridyl)-2,3-dihydro-5H-[1]benzopyrano[4,3c]pyridazin-3-one in 2 mL 1-methyl-2-pyrrolidone were added 55 mg Zn(CN)2 and 5 mg tetrakis(triphenylphosphine)palladium and stirred at 100.degree. for 1 h to give 34 mg 2-(2-cyanophenyl)-4-(3-pyridyl)-2,3-dihydro-5H-[1] benzopyrano [4,3-c] pyridazin-3-one (II). II inhibited the AMPA-induced influx of Ca into rat fetal cerebral cortex nerve cells with IC50 of 0.02 .mu.M.

RE.CNT 12 THERE ARE 12 CITED REFERENCES AVAILABLE FOR THIS RECORD ALL CITATIONS AVAILABLE IN THE RE FORMAT

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L12 ANSWER 3 OF 5 CAPLUS COPYRIGHT 2003 ACS on STN
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AN 2001:565039 CAPLUS

DN 135:153111

- TI Preparation of aryl-amidines and derivatives, and prodrugs thereof as factor Xa inhibitors
- IN Kang, Myung-Gyun; Park, Doo-Hee; Kwon, Oh-Hwan; Kim, Eunice Eun-Kyeong;
 Hwang, Kwang-Yeon; Heo, Yong-Seok; Park, Tae-Kyo; Lee, Tae-Hee; Moon,
 Kwang-Yul; Park, Jong-Woo; Chang, Hye-Kyung; Lee, Sang-Koo; Lee, Sun-Hwa;
 Park, Su-Kyung; Lee, Sung-Hack; Park, Hee-Dong
- PA LG Chem Investment Ltd., S. Korea
- SO PCT Int. Appl., 177 pp.

CODEN: PIXXD2

DT Patent

LA English

FAN.CNT 1

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PATENT NO.
                         KIND DATE
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OS

IT

RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)

(intermediate; prepn. of aryl-amidines and derivs., and prodrugs thereof as factor Xa inhibitors and anticoaqulants for treatment of thrombosis disorders)

RN150691-04-6 CAPLUS

CN Boronic acid, [2-[[(1,1-dimethylethyl)amino]sulfonyl]phenyl]- (9CI) (CA INDEX NAME)

RN 168618-42-6 CAPLUS

Boronic acid, [2-(methylthio)phenyl] - (9CI) (CA INDEX NAME) CN

183000-60-4 CAPLUS RN

CN Boronic acid, [2-[[(1,1-dimethylethyl)amino]sulfonyl]-5-methylphenyl]-(9CI) (CA INDEX NAME)

GI

$$Q = R^{1}$$

$$Q = R^{1}$$

$$R^{2}$$

$$Q = R^{1}$$

$$R^{2}$$

$$Q^{2} = R^{3}$$

$$Q^{3} = R^{3}$$

$$Q^{4} = R^{4}$$

$$Q^{6} = R^{4}$$

AB The aryl-amidines, particularly amidinoaryl-cyclopropanes, amidinoarylmethyl-pyrroles, amidinoaryl-benzenes, amidinoaryl-pyridines, or amindonoaryl-alanines, represented by formula G-A(D)-A-L-P[(X)n]-Q(Y)Z [wherein Ar = benzene, pyridine , thiophene, naphthalene, isoquinoline; G = R, F, Cl, Br, iodo, cyano, OR, O2CR, CO2R, CONR2 (wherein R = H, linear, branched, cyclic or branched cyclic C1-10 alkyl); A = Q-Q6, CH2 CHR5CONH, CH2CHR5CH2O, CH2CHR6NHCO [wherein R1, R2 = F, Cl, Br, iodo, R, CH2O R, CH2O2CR, CO2R, CONR2,

```
CON(CH2)m (m = 2-7), CO-morpholine, etc.; R3 = group listed in R2,
        CONH (amino acid or its ester or amide), etc.; R4 = F, Cl, Br, iodo, cyano,
        OR, R; R5 = NR2, NR(COR), NR (CH2)ml CO2R (m1 = 0-3), etc.; R6 = CO2R,
        CONR2, CH2OR]; Lb= CONH, CONHCH2, CH2NHCO, NHCONH, etc.; D = NH2, CH2NH2,
        C(:NR7)NH2 (wherein R7 = H, OH, CO2R8, OR8, O2COR8; wherein R8 = Ph,
        CH2Ph, linear, branched, cyclic or branched cyclic C1-10 alkyl); L =
         (CH2) m2 (m2 = 0,1); P = benzene, pyridine, pyrrole,
         furan, thiophene, oxazole, isoxazole, imidazole, 1,2-diazole, thiazole,
        isothiazole, pyridazine, pyridazine,
        pyrimidine, pyrazine, naphthalene, etc.; n = 0-2; Q = H,
        benzene, pyridine, pyridine, pyrrole, furan,
        thiophene, oxazole, isoxazole, imidazole, 1,2-diazole, thiazole,
        isothiazole, etc.; Y, Z = R, F, Cl, Br, iodo, cyano, OR, CO2R, COR, CONR2,
        NR2, NR(COR), N(COR)2, CF3, OCF3, etc.], pharmaceutically acceptable
        salts, prodrugs, hydrates, solvates or isomers thereof are prepd.
        compds. are inhibitors of coagulation enzyme, factor Xa (FXa). The
        present invention also relates to a pharmaceutical compn. contq. the above
        compd., and a method of using the same as an anticoagulant agent for
        treatment and prevention of thrombosis disorders. N-[4-(2-
        aminosulfonylphenyl) phenyl] -cis-2-(3-
        aminoiminomethylphenyl)cyclopropane-1-carboxamide monotrifluoroacetate,
        4-(4-aminoiminomethylbenzyl)-1-(3-aminoiminomethylbenzyl)pyrrole-3-
        carboxamide bis(trifluoroacetate), 3-aminoiminomethylbenzyl 2-(3-aminoiminomethylphenyl)benzyl ether bis(trifluoroacetate), and
         (S) - N - \{4 - (2 - aminosul fonylphenyl) benzoyl \} - 3 - (3 - aminosul fonylphenyl) benzoyl \} - 3 - (3 - aminosul fonylphenyl) benzoyl \} - 3 - (3 - aminosul fonylphenyl) benzoyl \} - 3 - (3 - aminosul fonylphenyl) benzoyl \} - 3 - (3 - aminosul fonylphenyl) benzoyl \} - 3 - (3 - aminosul fonylphenyl) benzoyl \} - 3 - (3 - aminosul fonylphenyl) benzoyl \} - 3 - (3 - aminosul fonylphenyl) benzoyl \} - 3 - (3 - aminosul fonylphenyl) benzoyl \} - 3 - (3 - aminosul fonylphenyl) benzoyl \} - 3 - (3 - aminosul fonylphenyl) benzoyl \} - 3 - (3 - aminosul fonylphenyl) benzoyl \} - 3 - (3 - aminosul fonylphenyl) benzoyl \} - 3 - (3 - aminosul fonylphenyl) benzoyl \} - 3 - (3 - aminosul fonylphenylphenyl) benzoyl \} - 3 - (3 - aminosul fonylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylphenylph
        aminoiminomethylphenyl)alanine Et ester trifluoroacetate in vitro
        inhibited FXa with Ki of 0.5, 0.12, 0.44, and 2 nM, resp., and thrombin
        with Ki of 2,900, 2.1, 5, and 620, resp., and exhibited the thrombin/FXa \,
        selectivity of 5,800, 18, 11, and 310, resp.
RE.CNT 4
                        THERE ARE 4 CITED REFERENCES AVAILABLE FOR THIS RECORD
                       ALL CITATIONS AVAILABLE IN THE RE FORMAT
L12 ANSWER 4 OF 5 CAPLUS COPYRIGHT 2003 ACS on STN
ΑN
        2000:457028 CAPLUS
DN
        133:89545
ΤI
        Substituted (aminoiminomethyl- or aminomethyl) benzoheteroaryl compounds
        useful as anticoaqulants
ΙN
        Dankulich, William P.; McGarry, Daniel G.; Burns, Christopher; Gallagher,
        Timothy F.; Volz, Francis A.
PΑ
        Aventis Pharmaceuticals Products Inc., USA
SO
        PCT Int. Appl., 215 pp.
        CODEN: PIXXD2
DT
        Patent
LΑ
        English
FAN.CNT 1
        PATENT NO.
                                     KIND DATE
                                                                         APPLICATION NO. DATE
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                      CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG
                                                                         US 1998-113710PA219981224
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					WO	1999-US	30623W	19991222		
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								119991222		
					US	2000-60)9103 A	120000630		
BERT	1D10 133 00E4E									

OS MARPAT 133:89545

IT 14047-29-1, 4-Carboxybenzeneboronic acid

RL: RCT (Reactant); RACT (Reactant or reagent)

(starting material; prepn. of substituted (aminoiminomethyl- or aminomethyl) benzoheteroaryl compds. as anticoagulants)

RN 14047-29-1 CAPLUS

CN Benzoic acid, 4-borono- (9CI) (CA INDEX NAME)

GI

$$R^2$$
 R^3
 NH_2
 $L^{1-Q-L^{2-R}}$

$$\operatorname{HN} = \bigvee_{\operatorname{NH}_2}^{\operatorname{H}} \bigcap_{\operatorname{H}} \bigcap_{\operatorname{II}}$$

AB The invention is directed to (aminoiminomethyl) - or (aminomethyl) - substituted benzoheteroaryl compds. I, which are useful as inhibitors of Factor Xa (no data) [wherein X = O, S, NH or derivs.; L1 = alkylene, alkenylene, alkynylene; L2 = bond, or as given for L1; Q = NH or derivs., O, CO, COO, OCO, NHCO or derivs., S(0)0-2, SO2NH or derivs, etc.; R = H, cycloalkyl, heterocyclyl, aryl, wide range of other cyclic groups; R2, R3 = H; or R2R3 = NH or derivs.]. The invention is also directed to compns. contg. the compds., methods for their prepn., and their use, e.g., in the inhibition of thrombin formation, or for treating a patient suffering from, or subject to, a disease state assocd. with excess thrombin. Approx. 160 examples were prepd. and claimed, and hundreds of intermediates were prepd. For instance, 4-(pyrid-3-yl)benzoic acid underwent amidation with 3-cyano-5-(2-aminoethyl)indole using TBTU and DIEA, and the product nitrile was treated with HCl(g) in MeOH followed by NH3 in MeOH, to give the invention compd. II.

L12 ANSWER 5 OF 5 CAPLUS COPYRIGHT 2003 ACS on STN

AN 1997:772291 CAPLUS

DN 128:48495

TI preparation of cyclic peptides as antifungal agents

IN Henle, Stacy Kay; Turner, William Wilson

PA Eli Lilly and Co., USA

SO U.S., 26 pp.

CODEN: USXXAM

DT Patent

LA English

FAN. CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
ΡI	US 5693611	Α	19971202	US 1997-785207	19970117
				US 1997-785207	19970117

OS MARPAT 128:48495

IT 136370-19-9P 146449-90-3P 158937-25-8P 194481-41-9P 194481-45-3P 194481-49-7P

RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)

(prepn. of cyclic peptides as antifungal agents)

10085368.1

Page 17

RN 136370-19-9 CAPLUS

CN Boronic acid, [4-(heptyloxy)phenyl] - (9CI) (CA INDEX NAME)

RN 146449-90-3 CAPLUS

CN Boronic acid, [4-(pentyloxy)phenyl]- (9CI) (CA INDEX NAME)

HO-B
$$O-(CH2)4-Me$$

RN 158937-25-8 CAPLUS

CN Boronic acid, [4'-(pentyloxy)[1,1'-biphenyl]-4-yl]- (9CI) (CA INDEX NAME)

Me- (CH₂)₄-0

$$B$$
-OH

OH

RN 194481-41-9 CAPLUS

CN Boronic acid, [6-[4-(pentyloxy)phenyl]-3-pyridinyl]- (9CI) (CA INDEX NAME)

RN 194481-45-3 CAPLUS

CN Boronic acid, [6-(heptyloxy)-3-pyridinyl]- (9CI) (CA INDEX NAME)

10085368.1

Page 18

OH
$$_{\text{HO-B}}$$
 $_{\text{N}}$ $_{\text{O- (CH}_2)_6-\text{Me}}$

RN 194481-49-7 CAPLUS

CN Boronic acid, [6'-(heptyloxy)[2,3'-bipyridin]-5-yl]- (9CI) (CA INDEX NAME)

GΙ

Cyclic peptides I [R' = H, Me, H2NCH2CH2, H2NCOCH2; R'', R''' = H, Me; Rx1 = H, NHR, OR (R = alkyl, benzyl, allyl, etc.); Rx2, Ry1, Ry2, Ry3, Ry4 = H, OH; R0 = OH, OP(O)(OH)2, OP(O)R1OH, OP(O)(OR1)OH (R1 = alkyl, Ph, benzyl, p-halo- or p-nitrophenyl or -benzyl), R2 = CO-A-X-B-Y-C-R3 (A, B, C = benzene, pyridine, pyridazine, pyrimidine, pyrazine, furan or thiophene ring; X, Y, = bond or

Ι

C.tplbond.C; R3 = alkyl, alkoxy)] or their pharmaceutically acceptable salts were prepd. as antifungal agents. Thus, I [R', R'', R''' = Me; Rx1, Rx2, Ry1, Ry2, Ry3, Ry4, R0 = OH, R2 = [6-[6-[4-(pentyloxy)phenyl]-3-pyridyl]-3-pyridyl] was prepd. tested against C. albicans (min. inhibitory concn. = 0.78 .mu.g/mL).

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            0 L4 AND FURANE
=> s 14 and methyl furane
             0 L4 AND METHYL FURANE
=> s 14 and thien
            74 L4 AND THIEN
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MISSING OPERATOR L7 FBIB
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The following are valid formats:
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APPS ----- AI, PRAI
BIB ----- AN, plus Bibliographic Data and PI table (default)
CAN ----- List of CA abstract numbers without answer numbers
CBIB ----- AN, plus Compressed Bibliographic Data
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IPC ----- International Patent Classifications
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             SCAN must be entered on the same line as the DISPLAY,
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HITRN ----- HIT RN and its text modification
HITSTR ----- HIT RN, its text modification, its CA index name, and
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HITSEQ ----- HIT RN, its text modification, its CA index name, its
             structure diagram, plus NTE and SEQ fields
FHITSTR ---- First HIT RN, its text modification, its CA index name, and
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KWIC ----- Hit term plus 20 words on either side
OCC ----- Number of occurrence of hit term and field in which it occurs
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All of the formats (except for SAM, SCAN, HIT, HITIND, HITRN, HITSTR, FHITSTR, HITSEQ, FHITSEQ, KWIC, and OCC) may be used with DISPLAY ACC to view a specified Accession Number. ENTER DISPLAY FORMAT (BIB):bib

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L17 ANSWER 1 OF 2 CAPLUS COPYRIGHT 2003 ACS on STN AN 2002:736258 CAPLUS DN 137:263048
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TI Imidazo-pyrimidine derivatives as ligands for GABA receptors, and their preparation, pharmaceutical compositions, and use in the

10085368.1 Page 21

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treatment of adverse neurological conditions.
     Chambers, Mark Stuart; Goodacre, Simon Charles; Hallett, David James;
IN
     Jennings, Andrew; Jones, Philip; Lewis, Richard Thomas; Moore, Kevin
     William; Russell, Michael Geoffrey Neil; Street, Leslie Joseph; Szekeres,
     Helen Jane
     Merck Sharp & Dohme Limited, UK
PA
     PCT Int. Appl., 197 pp.
SO
     CODEN: PIXXD2
DT
     Patent
     English
LΑ
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             ALL CITATIONS AVAILABLE IN THE RE FORMAT
     ANSWER 2 OF 2 CAPLUS COPYRIGHT 2003 ACS on STN
L17
AN
     2001:12273 CAPLUS
DN
     134:86271
ΤI
     Preparation of pyrimidine derivatives as Src-family protein
     tyrosine kinase inhibitor compounds
     Armstrong, Helen M.; Beresis, Richard; Goulet, Joung L.; Holmes, Mark A.;
IN
     Hong, Xingfang; Mills, Sander G.; Parsons, William H.; Sinclair, Peter J.;
     Steiner, Mark G.; Wong, Frederick; Zaller, Dennis M.
PA
     Merck & Co., Inc., USA
     PCT Int. Appl., 470 pp.
SO
     CODEN: PIXXD2
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     Patent
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     English
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                                           APPLICATION NO. DATE
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CAN ----- List of CA abstract numbers without answer numbers
CBIB ----- AN, plus Compressed Bibliographic Data
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FBIB ----- AN, BIB, plus Patent FAM
IND ----- Indexing data
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             e.g., D SCAN or DISPLAY SCAN)
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ISTD ----- STD, indented with text labels
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OIBIB ----- OBIB, indented with text labels
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SIBIB ----- IBIB, no citations
HIT ----- Fields containing hit terms
HITIND ----- IC, ICA, ICI, NCL, CC and index field (ST and IT)
             containing hit terms
HITRN ----- HIT RN and its text modification
HITSTR ----- HIT RN, its text modification, its CA index name, and
             its structure diagram
HITSEQ ----- HIT RN, its text modification, its CA index name, its
             structure diagram, plus NTE and SEQ fields
FHITSTR ---- First HIT RN, its text modification, its CA index name, and
             its structure diagram
FHITSEQ ---- First HIT RN, its text modification, its CA index name, its
             structure diagram, plus NTE and SEQ fields
KWIC ----- Hit term plus 20 words on either side
OCC ----- Number of occurrence of hit term and field in which it occurs
To display a particular field or fields, enter the display field
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To display a particular field or fields, enter the display field codes. For a list of the display field codes, enter HELP DFIELDS at an arrow prompt (=>). Examples of formats include: TI; TI,AU; BIB,ST; TI,IND; TI,SO. You may specify the format fields in any order and the information will be displayed in the same order as the format specification.

All of the formats (except for SAM, SCAN, HIT, HITIND, HITRN, HITSTR, FHITSTR, HITSEQ, FHITSEQ, KWIC, and OCC) may be used with DISPLAY ACC to view a specified Accession Number. ENTER DISPLAY FORMAT (BIB):bibn

'BIBN' IS NOT A VALID FORMAT FOR FILE 'CAPLUS'

The following are valid formats:

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ABS ----- GI and AB
ALL ------ BIB, AB, IND, RE
APPS ----- AI, PRAI
BIB ----- AN, plus Bibliographic Data and PI table (default)
CAN ----- List of CA abstract numbers without answer numbers
CBIB ----- AN, plus Compressed Bibliographic Data
DALL ----- ALL, delimited (end of each field identified)
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Page 24

FAM FBIB IND IPC MAX PATS SAM SCAN	MAX, delimited for post-processing AN, PI and PRAI in table, plus Patent Family data AN, BIB, plus Patent FAM Indexing data International Patent Classifications ALL, plus Patent FAM, RE PI, SO CC, SX, TI, ST, IT CC, SX, TI, ST, IT (random display, no answer numbers; SCAN must be entered on the same line as the DISPLAY, e.g., D SCAN or DISPLAY SCAN) BIB, IPC, and NCL			
IALL IBIB IMAX	ABS, indented with text labels ALL, indented with text labels BIB, indented with text labels MAX, indented with text labels STD, indented with text labels			
	AN, plus Bibliographic Data (original) OBIB, indented with text labels			
	BIB, no citations IBIB, no citations			
HITIND HITRN HITSTR	Fields containing hit terms IC, ICA, ICI, NCL, CC and index field (ST and IT) containing hit terms HIT RN and its text modification HIT RN, its text modification, its CA index name, and its structure diagram HIT RN, its text modification, its CA index name, its structure diagram, plus NTE and SEQ fields			
FHITSTR	First HIT RN, its text modification, its CA index name, and			
KWIC	its structure diagram First HIT RN, its text modification, its CA index name, its structure diagram, plus NTE and SEQ fields Hit term plus 20 words on either side Number of occurrence of hit term and field in which it occurs			
To display a particular field or fields, enter the display field				

To display a particular field or fields, enter the display field codes. For a list of the display field codes, enter HELP DFIELDS at an arrow prompt (=>). Examples of formats include: TI; TI,AU; BIB,ST; TI,IND; TI,SO. You may specify the format fields in any order and the information will be displayed in the same order as the format specification.

All of the formats (except for SAM, SCAN, HIT, HITIND, HITRN, HITSTR, FHITSTR, HITSEQ, FHITSEQ, KWIC, and OCC) may be used with DISPLAY ACC to view a specified Accession Number. ENTER DISPLAY FORMAT (BIB):BIB

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L19 ANSWER 1 OF 2 CAPLUS COPYRIGHT 2003 ACS on STN
```

AN 2002:736258 CAPLUS

DN 137:263048

TI Imidazo-pyrimidine derivatives as ligands for GABA receptors, and their preparation, pharmaceutical compositions, and use in the treatment of adverse neurological conditions.

```
Chambers, Mark Stuart; Goodacre, Simon Charles; Hallett, David James;
IN
    Jennings, Andrew; Jones, Philip; Lewis, Richard Thomas; Moore, Kevin
    William; Russell, Michael Geoffrey Neil; Street, Leslie Joseph; Szekeres,
    Helen Jane
PA
    Merck Sharp & Dohme Limited, UK
SO
    PCT Int. Appl., 197 pp.
     CODEN: PIXXD2
DT
     Patent
LΑ
    English
FAN.CNT 3
     PATENT NO.
                     KIND DATE
                                           APPLICATION NO.
                                                           DATE
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                            20020926
PΙ
    WO 2002074773
                      A1
                                          WO 2002-GB1352
                                                            20020319
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            LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NO, NZ, OM, PH, PL,
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            BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG
                                         US 2002-100797 20020319
     US 2002193385
                      -A1
                            20021219
PRAI GB 2001-7134
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                            20010321
     GB 2001-27938
                      Α
                            20011121
     US 2000-719712
                      A3
                            20001215
     MARPAT 137:263048
OS
             THERE ARE 2 CITED REFERENCES AVAILABLE FOR THIS RECORD
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    ANSWER 2 OF 2 CAPLUS COPYRIGHT 2003 ACS on STN
L19
     2001:12273 CAPLUS
ΑN
DN
     134:86271
     Preparation of pyrimidine derivatives as Src-family protein
ΤI
     tyrosine kinase inhibitor compounds
IN
     Armstrong, Helen M.; Beresis, Richard; Goulet, Joung L.; Holmes, Mark A.;
     Hong, Xingfang; Mills, Sander G.; Parsons, William H.; Sinclair, Peter J.;
     Steiner, Mark G.; Wong, Frederick; Zaller, Dennis M.
PA
     Merck & Co., Inc., USA
     PCT Int. Appl., 470 pp.
SO
     CODEN: PIXXD2
DT
     Patent
LΑ
     English
FAN.CNT 1
     PATENT NO.
                      KIND DATE
                                           APPLICATION NO.
                                                            DATE
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     WO 2001000213
                      A1
                            20010104
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    US 6498165 B1 20021224 US 2000-604305 20000626
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PRAI US 1999-141639P P
                          19990630
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                        20000626
    WO 2000-US17443
OS MARPAT 134:86271
RE.CNT 4 THERE ARE 4 CITED REFERENCES AVAILABLE FOR THIS RECORD
             ALL CITATIONS AVAILABLE IN THE RE FORMAT
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L3
         5071 S L1 SSS FULL
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          8602 S L3
L4
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L5
L6
           0 S L4 AND TOULENE
L7
          2038 S L4 AND PHENYL
L8
           308 S L5 AND L7
          1081 S L4 AND PYRIDINE
L9
          332 S L4 AND PYRIMIDINE
L10
L11
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L12
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=> s 121 and 122 and 123 and 124
L26 0 L21 AND L22 AND L23 AND L24
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10085368.1 Page 27

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L26 HAS NO ANSWERS
'FBIB HITSTR ABS ' IS NOT A VALID STRUCTURE FORMAT KEYWORD
Structure Formats
SIA ---- Structure Image, Attributes, and map table if it contains
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SIM ---- Structure IMage.
SAT ---- Structure ATtributes and map table if it contains data.
SCT ---- Structure Connection Table and map table if it contains
          data.
SDA ---- All Structure DAta (image, attributes, connection table and
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NOS ---- NO Structure data.
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L25 ANSWER 1 OF 8 CAPLUS COPYRIGHT 2003 ACS on STN
     2003:610410 CAPLUS
AN
DN
     139:179889
TI
    Methylene amides, particularly [(arylmethyl)amino](oxo)acetic acids,
    useful as modulators, and especially inhibitors, of protein tyrosine
    phosphatases (PTPs), and their preparation, uses, e.g., as antidiabetics,
     and pharmaceutical compositions.
     Swinnen, Dominique; Bombrun, Agnes; Gonzalez, Jerome; Gerber, Patrick;
IN
     Pittet, Pierre-Andre
     Applied Research Systems ARS Holding N.V., Neth. Antilles
PA
SO
     PCT Int. Appl., 346 pp.
     CODEN: PIXXD2
DT
     Patent
LΑ
     English
FAN.CNT 1
                     KIND DATE
     PATENT NO.
                                          APPLICATION NO. DATE
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PΙ
     WO 2003064376
                     A1
                            20030807
                                          WO 2003-EP808 20030127
         W: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN,
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             PL, PT, RO, RU, SC, SD, SE, SG, SK, SL, TJ, TM, TN, TR, TT, TZ,
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             RU, TJ, TM
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             ML, MR, NE, SN, TD, TG
                                           EP 2002-100078 A 20020129
                                           EP 2002-100410 A 20020425
IT
     5720-05-8, 4-Tolylboronic acid 100124-06-9,
     Dibenzofuran-4-boronic acid
     RL: RCT (Reactant); RACT (Reactant or reagent)
        (starting material; prepn. of [(arylmethyl)amino](oxo)acetic acids as
        PTP inhibitors for use as antidiabetics, etc.)
     5720-05-8 CAPLUS
RN
CN
     Boronic acid, (4-methylphenyl) - (9CI) (CA INDEX NAME)
```

RN 100124-06-9 CAPLUS

CN Boronic acid, 4-dibenzofuranyl- (9CI) (CA INDEX NAME)

GΙ

AB Title compds. I [wherein R1 = alkyl, alkenyl, alkynyl, aryl, heteroaryl, (3-8-membered)-cycloalkyl, heterocycloalkyl, (alkyl)aryl, (alkyl)heteroaryl, (alkenyl)aryl, heteroaryl, (alkynyl)aryl, heteroaryl; R2, R3 = independently H or alkyl; Cy = aryl, heteroaryl, cycloalkyl, heterocyclyl; with the proviso that four compds. are excluded; their geometrical isomers, optically active forms as enantiomers, diastereomers

and racemates, and pharmaceutically acceptable salts and active derivs. were prepd. as inhibitors of protein tyrosine phosphatases (PTPs), in particular PTP1B. Examples include over 400 invention compds., five pharmaceutical formulations, and two biol. assays. For example, II was prepd. in 4 steps by amidation of 4-formylbenzoic acid with dodecylamine in THF in the presence of 4-methylmorpholine and iso-Bu chloroformate for 3 h at room temp., reductive amination with 4-trifluoromethylbenzylamine in DCE in the presence of NaBH(OAc)3, TEA-acylation with chlorooxoacetic acid Et ester in THF, and base-catalyzed hydrolysis of the ester. II exhibited an IC50 value of 2.224 .mu.M for inhibition of PTP1B, 1.40 .mu.M for GLEPP-1, 2.40 .mu.M for SHP-1, and 2.70 .mu.M for SHP-2 in an in vitro assay. In an in vivo postprandial glycemia model in db/db mice, II, at 20-200 mg/kg orally, decreased blood glucose level by 17% at 20 mg/kg, by 42% at 100 mg/kg, and by 48% at 200 mg/kg, with decreases in serum insulin levels of -2%, 66%, and 89%, resp. Thus, I and their formulations are useful for the treatment and/or prevention of metabolic disorders mediated by insulin resistance or hyperglycemia, comprising diabetes type I and/or II, inadequate glucose tolerance, insulin resistance, hyperlipidemia, hypertriglyceridemia, hypercholesterolemia, obesity, polycystic ovary syndrome (PCOS).

RE.CNT 7 THERE ARE 7 CITED REFERENCES AVAILABLE FOR THIS RECORD ALL CITATIONS AVAILABLE IN THE RE FORMAT

```
ANSWER 2 OF 8 CAPLUS COPYRIGHT 2003 ACS on STN
    2003:472495 CAPLUS
AN
DN
    139:53008
TI
    Preparation of benzoxazoles and analogues as estrogenic agents
IN
    Malamas, Michael Sotirios; McDvitt, Robert Emmett; Gunawan, Iwan; Manas,
    Eric Steven; Collini, Michael David; Harris, Heather Anne; Keith, James
    Carl, Jr.; Albert, Leo Massillamoney; Lyttle, Cecil Richard
PΑ
    Wyeth, John, and Brother Ltd., USA
SO
    PCT Int. Appl., 100 pp.
    CODEN: PIXXD2
DT
    Patent
    English
LΑ
FAN.CNT 1
    PATENT NO.
                    KIND DATE
                                      APPLICATION NO. DATE
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PΙ
    WO 2003050095
                    A1
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                                      WO 2002-US38513 20021203
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            MR, NE, SN, TD, TG
                                       US 2001-336663PP 20011205
OS
    MARPAT 139:53008
ΙT
    98-80-6, Benzeneboronic acid
    RL: RCT (Reactant); RACT (Reactant or reagent)
       (prepn. of benzoxazoles and analogs as ER-.alpha. and ER-.beta.
       modulators)
RN
    98-80-6 CAPLUS
CN
    Boronic acid, phenyl- (9CI) (CA INDEX NAME)
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GI

$$\begin{array}{c|c} & & & & \\ & & & \\ R2 & & & \\ \hline & & & \\ R1 & & & \\ \hline & & & \\ & & & \\ & & & \\ & & & \\ & & & \\ & & & \\ & & & \\ & & & \\ & & & \\ & & & \\ & & & \\ & & & \\ & & & \\ & & & \\ & & & \\ & & & \\ & & \\ & & & \\ &$$

AΒ Title compds. I [wherein R1 = (un) substituted alkenyl; R2 and R2a = independently H, OH, halo, alkoxy, trifluoroalkyl, trifluoroalkoxy, or (un) substituted alkyl, alkenyl, or alkynyl; R3 and R3a = independently H, halo, alkoxy, trifluoroalkyl, trifluoroalkoxy, or (un) substituted alkyl, alkenyl, or alkynyl; R5 and R6 = independently H, alkyl, or aryl; X = O, S, or NR7; R7 = H, alkyl, aryl, COR5, CO2R5, or SO2R5; and pharmaceutically acceptable salts thereof] were prepd. as estrogen receptor (ER) modulators. For example, 2,5-dimethoxybenzoic acid was condensed with 2,5-dimethoxyaniline using thionyl chloride in THF to give the amide (93%). Reaction of the amide with pyridine.bul.HCl at 200.degree. for 1 h afforded the benzoxazole II (76%). I exhibited binding affinities for ER-.alpha. and ER-.beta. in the ranges of 44 nM to >10,000 nM and 1 nM to 1600 nM, resp., indicating that these compds. are preferentially ER-.beta. selective ligands but are still active at ER-.alpha.. Selected invention compds. were also tested for metallothionein II mRNA and progesterone mRNA regulation, uterotropic activity, osteoporosis and lipid modulation, estrogen receptor antagonist activity via the rat hot flush test, mammotrophic activity, inflammatory bowel disease treatment, antiarthritic activity, neuroprotection, ovulation inhibition, and endometriosis treatment. Thus, I and pharmaceutical compns. thereof are useful for the treatment of a wide variety of ER-assocd. conditions and diseases.

RE.CNT 5 THERE ARE 5 CITED REFERENCES AVAILABLE FOR THIS RECORD ALL CITATIONS AVAILABLE IN THE RE FORMAT

L25 ANSWER 3 OF 8 CAPLUS COPYRIGHT 2003 ACS on STN

AN 2003:334903 CAPLUS

DN 138:353988

TI Preparation of benzimidazoles and analogs and their use as protein kinase inhibitors

IN Edwards, Michael Louis; Cox, Paul Joseph; Amendola, Shelley; Deprets,

```
Stephanie Daniele; Gillespy, Timothy Alan; Edlin, Christopher David;
     Morley, Andrew David; Gardner, Charles J.; Pedgrift, Brian; Bouchard,
     Herve; Babin, Didier; Gauzy, Laurence; Le Brun, Alain; Majid, Tahir
     Nedeem; Reader, John C.; Payne, Lloyd J.; Khan, Nawaz M.; Cherry, Michael
PA
     Aventis Pharmaceuticals Inc., USA
SO
     PCT Int. Appl., 711 pp.
     CODEN: PIXXD2
DT
     Patent
     English
LA
FAN.CNT 1
     PATENT NO.
                      KIND DATE
                                           APPLICATION NO.
                                                            DATE
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                            20030501
PΤ
    WO 2003035065
                      Α1
                                           WO 2002-GB4763
                                                            20021024
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             GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR,
             LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NO, NZ, OM, PH,
             PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TN, TR, TT, TZ,
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             NE, SN, TD, TG
                                           FR 2001-13868 A 20011026
                                           GB 2002-6893
                                                          A 20020322
                                           GB 2002-6895
                                                          A 20020322
                                           US 2002-395060PP 20020711
                                           US 2002-395151PP 20020711
     FR 2831537
                       A1 . 20030502
                                           FR 2001-13868
                                                            20011026
OS
    MARPAT 138:353988
IT
     98-80-6, Phenylboronic acid 1423-26-3,
     (3-(Trifluoromethyl)phenyl)boronic acid 1679-18-1,
     4-Chlorophenylboronic acid 1692-25-7, Pyridine-3-boronic acid
     1765-93-1, (4-Fluorophenyl)boronic acid 1993-03-9,
     2-Fluorophenylboronic acid 5122-94-1, (Biphenyl-4-yl)boronic
     acid 5720-05-8, 4-Methylphenylboronic acid 5720-06-9,
     2-Methoxyphenylboronic acid 5720-07-0, (4-Methoxyphenyl)boronic
    acid 10365-98-7, (3-Methoxyphenyl)boronic acid
     13922-41-3, (1-Naphthyl)boronic acid 16152-51-5,
     (4-Isopropylphenyl)boronic acid 16419-60-6, (2-Tolyl)boronic
     acid 17933-03-8, (3-Tolyl)boronic acid 55499-43-9,
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    pheny1)boronic acid 63503-60-6, (3-Chlorophenyl)boronic
     acid 68716-47-2, (2,4-Dichlorophenyl)boronic acid
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     (3-(Hydroxymethyl)phenyl)boronic acid 87199-18-6,
     (3-Hydroxyphenyl)boronic acid 94839-07-3, 3,4-
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     (Thianthren-1-yl)boronic acid 122775-35-3, (3,4-
    Dimethoxyphenyl)boronic acid 123324-71-0, (4-tert-
    Butylphenyl)boronic acid 128796-39-4, (4-(Trifluoromethyl)
    phenyl)boronic acid 139301-27-2, (4-(Trifluoromethoxy)
    phenyl)boronic acid 139911-29-8, (4-Fluoro-2-
     methylphenyl)boronic acid 144025-03-6, (2,4-
    Difluorophenyl)boronic acid 144432-85-9, (3-Chloro-4-
     fluorophenyl)boronic acid 145349-76-4, (4-(Ethylthio)
    phenyl)boronic acid 146631-00-7, (4-(Benzyloxy)
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phenyl)boronic acid 149104-88-1, (4-(Methylsulfonyl) phenyl)boronic acid 149104-90-5, (4-Acetylphenyl)boronic acid 150255-96-2, 3-Cyanophenylboronic acid 151169-75-4 , (3,4-Dichlorophenyl)boronic acid 156682-54-1, (3-(Benzyloxy) phenyl) boronic acid 166316-48-9, (4-(3-Hydroxy-3oxopropyl) phenyl) boronic acid 172975-69-8, (3,5-Dimethylphenyl)boronic acid 178305-99-2, (3-Fluoro-4-phenylphenyl)boronic acid 179113-90-7, (3-(Trifluoromethoxy)phenyl)boronic acid 182163-96-8, (3,4,5-Trimethoxyphenyl)boronic acid 204841-19-0, (3-Acetylphenyl)boronic acid RL: RCT (Reactant); RACT (Reactant or reagent) (prepn. of benzimidazoles and analogs and their use as protein kinase inhibitors) 98-80-6 CAPLUS RNBoronic acid, phenyl- (9CI) (CA INDEX NAME) CN

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RN 1423-26-3 CAPLUS CN Boronic acid, [3-(trifluoromethyl)phenyl]- (9CI) (CA INDEX NAME)

RN 1679-18-1 CAPLUS CN Boronic acid, (4-chlorophenyl)- (9CI) (CA INDEX NAME)

RN 1692-25-7 CAPLUS CN Boronic acid, 3-pyridinyl- (9CI) (CA INDEX NAME)

RN 1765-93-1 CAPLUS

CN Boronic acid, (4-fluorophenyl) - (9CI) (CA INDEX NAME)

RN 1993-03-9 CAPLUS

CN Boronic acid, (2-fluorophenyl) - (9CI) (CA INDEX NAME)

RN 5122-94-1 CAPLUS

CN Boronic acid, [1,1'-biphenyl]-4-yl- (9CI) (CA INDEX NAME)

RN 5720-05-8 CAPLUS

CN Boronic acid, (4-methylphenyl) - (9CI) (CA INDEX NAME)

RN 5720-06-9 CAPLUS

CN Boronic acid, (2-methoxyphenyl) - (9CI) (CA INDEX NAME)

RN 5720-07-0 CAPLUS

CN Boronic acid, (4-methoxyphenyl) - (9CI) (CA INDEX NAME)

RN 10365-98-7 CAPLUS

CN Boronic acid, (3-methoxyphenyl) - (9CI) (CA INDEX NAME)

RN 13922-41-3 CAPLUS

CN Boronic acid, 1-naphthalenyl- (9CI) (CA INDEX NAME)

RN 16152-51-5 CAPLUS

CN Boronic acid, [4-(1-methylethyl)phenyl]- (9CI) (CA INDEX NAME)

RN 16419-60-6 CAPLUS

CN Boronic acid, (2-methylphenyl) - (9CI) (CA INDEX NAME)

RN 17933-03-8 CAPLUS

CN Boronic acid, (3-methylphenyl) - (9CI) (CA INDEX NAME)

RN 55499-43-9 CAPLUS

CN Boronic acid, (3,4-dimethylphenyl) - (9CI) (CA INDEX NAME)

RN 59016-93-2 CAPLUS

CN Boronic acid, [4-(hydroxymethyl)phenyl]- (9CI) (CA INDEX NAME)

RN 63503-60-6 CAPLUS

CN Boronic acid, (3-chlorophenyl) - (9CI) (CA INDEX NAME)

RN 68716-47-2 CAPLUS

CN Boronic acid, (2,4-dichlorophenyl) - (9CI) (CA INDEX NAME)

RN 71597-85-8 CAPLUS

CN Boronic acid, (4-hydroxyphenyl) - (9CI) (CA INDEX NAME)

RN 87199-15-3 CAPLUS

CN Boronic acid, [3-(hydroxymethyl)phenyl]- (9CI) (CA INDEX NAME)

RN 87199-18-6 CAPLUS

CN Boronic acid, (3-hydroxyphenyl) - (9CI) (CA INDEX NAME)

RN 94839-07-3 CAPLUS

CN Boronic acid, 1,3-benzodioxol-5-yl- (9CI) (CA INDEX NAME)

Patel

9/24/2003>

RN 108847-76-3 CAPLUS

CN Boronic acid, 1-thianthrenyl- (9CI) (CA INDEX NAME)

.RN 122775-35-3 CAPLUS

CN Boronic acid, (3,4-dimethoxyphenyl) - (9CI) (CA INDEX NAME)

RN 123324-71-0 CAPLUS

CN Boronic acid, [4-(1,1-dimethylethyl)phenyl]- (9CI) (CA INDEX NAME)

Patel

RN 128796-39-4 CAPLUS

CN Boronic acid, [4-(trifluoromethyl)phenyl]- (9CI) (CA INDEX NAME)

RN 139301-27-2 CAPLUS

CN Boronic acid, [4-(trifluoromethoxy)phenyl] - (9CI) (CA INDEX NAME)

RN 139911-29-8 CAPLUS

CN Boronic acid, (4-fluoro-2-methylphenyl) - (9CI) (CA INDEX NAME)

RN 144025-03-6 CAPLUS

CN Boronic acid, (2,4-difluorophenyl) - (9CI) (CA INDEX NAME)

RN 144432-85-9 CAPLUS

CN Boronic acid, (3-chloro-4-fluorophenyl) - (9CI) (CA INDEX NAME)

RN 145349-76-4 CAPLUS

CN Boronic acid, [4-(ethylthio)phenyl]- (9CI) (CA INDEX NAME)

RN 146631-00-7 CAPLUS

CN Boronic acid, [4-(phenylmethoxy)phenyl]- (9CI) (CA INDEX NAME)

RN 149104-88-1 CAPLUS

CN Boronic acid, [4-(methylsulfonyl)phenyl]- (9CI) (CA INDEX NAME)

RN 149104-90-5 CAPLUS

CN Boronic acid, (4-acetylphenyl) - (9CI) (CA INDEX NAME)

Patel

RN 150255-96-2 CAPLUS

CN Boronic acid, (3-cyanophenyl) - (9CI) (CA INDEX NAME)

RN 151169-75-4 CAPLUS

CN Boronic acid, (3,4-dichlorophenyl) - (9CI) (CA INDEX NAME)

RN 156682-54-1 CAPLUS

CN Boronic acid, [3-(phenylmethoxy)phenyl] - (9CI) (CA INDEX NAME)

RN 166316-48-9 CAPLUS

CN Benzenepropanoic acid, 4-borono- (9CI) (CA INDEX NAME)

RN 172975-69-8 CAPLUS

CN Boronic acid, (3,5-dimethylphenyl) - (9CI) (CA INDEX NAME)

RN 178305-99-2 CAPLUS

CN Boronic acid, (2-fluoro[1,1'-biphenyl]-4-yl)- (9CI) (CA INDEX NAME)

RN 179113-90-7 CAPLUS

CN Boronic acid, [3-(trifluoromethoxy)phenyl]- (9CI) (CA INDEX NAME)

RN 182163-96-8 CAPLUS

CN Boronic acid, (3,4,5-trimethoxyphenyl) - (9CI) (CA INDEX NAME)

RN 204841-19-0 CAPLUS

CN Boronic acid, (3-acetylphenyl) - (9CI) (CA INDEX NAME)

GI

AΒ The invention is directed to physiol, active benzimidazoles and analogs (shown as I; variables defined below; e.g. 2-(1H-indazol-3-yl)-1Hbenzimidazole-5-carboxylic acid benzylamide) and compns. contg. such compds., and their prodrugs, and pharmaceutically acceptable salts and solvates of such compds. and their prodrugs, as well as to novel I and to processes for their prepn. Such compds. and compns. have valuable pharmaceutical properties, in particular the ability to inhibit kinases. For I: X = C-R2 and W, Y and Z = CH or CR3; or W = CH, X = N, Y = CH or CR3, and Z = CH or CR3; or W = N, X = CH or CR2, Y = CH and CR3, and Z =CH or CR3; or W = N, X = CH or CR2, Y = N, and Z is CH or CR3; or W = N, X= CH or CR2, Y = CH or CR3, and Z = N; or W = N, X = N, Y = CH or CR3, and Z = CH or CR3. A5 = H or alkyl; R1 = optionally substituted aryl or heteroaryl; addnl. details are given in the claims. IC50 values for >200 I are tabulated for inhibition of KDR receptor tyrosine kinase. Particular I inhibit SYK activity with IC50's = $100 \, .mu.M$ to $0.1 \, nM.$ Particular I inhibit ITK activity with IC50's = 100 .mu.M to 1 .mu.M. I inhibit the increase in edema obsd. in a sensitized mouse ear following antigen exposure and inhibit mast cell activation and functional responses when given orally in a mouse model of passive cutaneous anaphylaxis. Methods of prepn. are claimed and hundreds of example prepns. of I and intermediates leading to them are included. For example, 20 mg 2-(1H-indazol-3-yl)-1H-benzimidazole-5-carboxylic acid benzylamide was prepd. from 20 mg 2-(1H-indazol-3-yl)-1H-benzimidazole-5-carboxylic acid and benzylamine in DMF in the presence of HBTU followed by addn. of N, N-diisopropylethylamine; the acid was prepd. in several steps starting from 3-indazolecarboxylic acid and involving intermediates Me 3-indazolecarboxylate, (1H-indazol-3-yl)methanol and 1H-indazole-3carboxaldehyde.

RE CNT 20 THERE ARE 20 CITED REFERENCES AVAILABLE FOR THIS RECORD ALL CITATIONS AVAILABLE IN THE RE FORMAT

L25 ANSWER 4 OF 8 CAPLUS COPYRIGHT 2003 ACS on STN

AN 2003:242160 CAPLUS

DN 138:271705

TI Preparation of triazinyl and other carboxamides as inhibitors of histone deacetylase

9/24/2003>

PA Methylgene, Inc., Can.

Patel

FAN.CNT 1

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SO PCT Int. Appl., 347 pp.
CODEN: PIXXD2
DT Patent
LA English
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	PAT	ENT	NO.		KIND DATE					APPLICATION NO. DATE								
ΡI	WO 2003024448				 A:	 2	2003	0327		WO 2002-US29017 20020912								
		W :	ΑE,	AG,	AL,	AM,	AT,	AU,	ΑZ,	ΒA,	BB,	BG,	BR,	BY,	ΒZ,	CA,	CH,	CN,
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			GM,	HR,	HU,	ID,	IL,	IN,	IS,	JP,	ΚE,	KG,	KP,	KR,	KZ,	LC,	LK,	LR,
			LS,	LT,	LU,	LV,	MA,	MD,	MG,	MK,	MN,	MW,	MX,	MZ,	NO,	NZ,	OM,	PH,
			PL,	PT,	RO,	RU,	SD,	SE,	SG,	SI,	SK,	SL,	TJ,	TM,	TN,	TR,	TT,	TZ,
			UA,	UG,	US,	UΖ,	VN,	YU,	ZA,	ZM,	ZW,	AM,	AZ,	BY,	KG,	ΚZ,	MD,	RU,
			TJ,	TM														
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			CH,	CY,	CZ,	DE,	DK,	EE,	ES,	FI,	FR,	GB,	GR,	ΙE,	ΙT,	LU,	MC,	NL,
			PT,	SE,	SK,	TR,	BF,	ВJ,	CF,	CG,	CI,	CM,	GA,	GN,	GQ,	GW,	ML,	MR,
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US 2001-322402PP 20010914 US 2002-391728PP 20020626

OS MARPAT 138:271705

IT 10365-98-7, 3-Methoxyphenylboronic acid

RL: RCT (Reactant); RACT (Reactant or reagent)

(prepn. of triazinyl and other carboxamides as inhibitors of histone deacetylase for treating cell proliferative disorders)

RN 10365-98-7 CAPLUS

CN Boronic acid, (3-methoxyphenyl) - (9CI) (CA INDEX NAME)

GI

AB The invention relates to triazines (shown as I; variables defined below; e.g. 4-[[4-amino-6-(2-indanylamino)-[1,3,5]triazin-2-ylamino]methyl]-N-(2-aminophenyl)benzamide) and Cy3-X1-Ar2-(C(R5):C(R6))qC(O)NH-Ay2 (II; variables defined below; e.g.), many of which are N-(o-aminophenyl)carboxamides, as inhibitors of histone deacetylase (data included for many I and II). The invention provides compds. and methods for inhibiting histone deacetylase enzymic activity. The invention also provides compns. and methods for treating cell proliferative diseases and

conditions. Antineoplastic effects of some I and II are illustrated for colorectal, pulmonary and pancreatic neoplasms; also the combined antineoplastic effect of histone deacetylase inhibitors and histone deacetylase antisense oligonucleotides on tumor cells in vivo was demonstrated. For I: R3 and R4 = H, L1, Cy1 and -L1-Cy1 (L1 = C1-C6 alkyl, C2-C6 heteroalkyl, or C3-C6 alkenyl; Cy1 = cycloalkyl, aryl, heteroaryl, or heterocyclyl) or R3 and R4 are taken together with the adjacent N atom to form a 5-, 6-, or 7-membered ring, wherein the ring atoms = C, O, S, and N, and wherein the ring is optionally substituted, and optionally forms part of a bicyclic ring system, or is optionally fused to one or two aryl or heteroaryl rings, or to one or two satd. or partially unsatd. cycloalkyl or heterocyclic rings, each of which rings and ring systems is optionally substituted. Y1 = -N(R1)(R2), -CH2-C(0)-N(R1)(R2), halogen, and H (R1 and R2 = H, L1, Cy1, and -L1-Cy1). Y2 = chem. bond or N(R0) (R0 = H, alkyl, aryl, aralkyl, and acyl); Ak1 =C1-C6 alkylene, C1-C6-heteroalkylene (preferably, in which one -CH2- is replaced with -NH-, and more preferably -NH-CH2), C2-C6 alkenylene or C2-C6 alkynylene; Ar1 = arylene or heteroarylene, either of which is optionally substituted; and Z1 = C(0)NH-Ay1 and CH:CHC(0)NH-Ay1 (Ay1 = aryl or heteroaryl, each of which is optionally substituted). For II: Cy2 = cycloalkyl, aryl, heteroaryl, or heterocyclyl; X1 = covalent bond, M1-L2-M1, and L2-M2-L2 (L2 = chem. bond, C1-C4 alkylene, C2-C4 alkenylene, and C2-C4 alkynylene, provided that L2 is not a chem. bond when X1 is M1-L2-M1; M1 = -O-, -N(R7)-, -S-, -S(O)-, S(O)2-, -S(O)2N(R7)-, -N(R7)S(O)2-, -C(O)-, -C(O)NH-, -NHC(O)-, -NHC(O)-O- and -OC(O)NH- (R7 = H, alkyl, aryl, aralkyl, acyl, heterocyclyl, and heteroaryl); and M2 = M1, heteroarylene, and heterocyclylene, either of which rings is optionally substituted). Ar2 = arylene or heteroarylene, each of which is optionally substituted; R5 and R6 = H, alkyl, aryl, and aralkyl; q is 0 or 1; and Ay2 is a 5-6 membered cycloalkyl, heterocyclyl, or heteroaryl substituted with an amino or hydroxy moiety (preferably these groups are ortho to the amide N to which Ay2 is attached) and further optionally substituted; provided that when Cy2 is naphthyl, X1 is -CH2-, Ar2 is Ph, R5 and R6 are H, and q is 0 or 1, Ay2 is not Ph or o-hydroxyphenyl. Although the methods of prepn. are not claimed, hundreds of example prepns. are included.

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L25 ANSWER 5 OF 8 CAPLUS COPYRIGHT 2003 ACS on STN
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FAN. CNT 3

	C111 J				
	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
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				US 1999-405524 A	A319990923
	US 6448295	B1	20020910	US 2001-991208	20011114

AN 2002:658752 CAPLUS

DN 137:201139

TI Substituted polycyclic aryl and heteroaryl tertiary-heteroalkylamines useful for inhibiting cholesteryl ester transfer protein activity

IN Sikorski, James A.; Durley, Richard C.; Mischke, Deborah A.; Reinhard, Emily J.; Fobian, Yvette M.; Tollefson, Michael B.; Wang, Lijuan; Grapperhaus, Margaret L.; Hickory, Brian S.; Massa, Mark A.; Norton, Monica B.; Vernier, William F.; Parnas, Barry L.; Promo, Michele A.; Hamme, Ashton T.; Spangler, Dale P.; Rueppel, Melvin L.

PA USA

SO U.S. Pat. Appl. Publ., 157 pp., Division of U.S. Ser. No. 405,524. CODEN: USXXCO

DT Patent

LA English

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		C4534	000				2002	0017				999-4						
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	US	64588	849		B	Ţ	2002	T00T				001-9			2001			
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     PATENT NO.
                     KIND DATE
                                          APPLICATION NO. DATE
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    WO 2000018724
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            AE, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CR, CU,
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    JP 2002525351
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                                           US 1998-101663PP 19980925
                                        · WO 1999-US22120W 19990923
OS
    MARPAT 137:201139
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98-80-6, Phenylboronic acid 1423-26-3, 3-Trifluoromethyl benzeneboronic acid 1423-27-4, 2-(Trifluoromethyl)phenylboronic acid 87199-16-4, 3-Formylphenylboronic acid RL: RCT (Reactant); RACT (Reactant or reagent) (reactant; prepn. of substituted polycyclic aryl and heteroaryl tertiary-heteroalkylamines as cholesteryl ester transfer protein inhibitors for the treatment of atherosclerosis and other coronary artery disease)

RN 98-80-6 CAPLUS

CN Boronic acid, phenyl- (9CI) (CA INDEX NAME)

1423-26-3 CAPLUS RN

Boronic acid, [3-(trifluoromethyl)phenyl] - (9CI) (CA INDEX NAME) CN

1423-27-4 CAPLUS RN

Boronic acid, [2-(trifluoromethyl)phenyl] - (9CI) (CA INDEX NAME) CN

RN 87199-16-4 CAPLUS

Boronic acid, (3-formylphenyl) - (9CI) (CA INDEX NAME) CN

GI

* STRUCTURE DIAGRAM TOO LARGE FOR DISPLAY - AVAILABLE VIA OFFLINE PRINT *

Title compds. I [X = NH, N(OH), N-alkyl; R16 = hydrido; n = 1-2; R1 =AB haloalkyl, haloalkoxyalkyl; R2 = hydrido, hydroxyalkyl, aryl, aralkyl, alkyl, alkenyl, alkynyl, etc.; R3 = hydrido, alkyl, alkenyl, alkoxyalkyl, aryl, aralkyl, heteroaryl, heteroaralkyl, alkenyloxyalkyl, etc.; Y = bond, alkyl; Z = bond, alkyl; R4, R8-9, R13 = hydrido, halo, haloalkyl, alkyl; R5-7, R10-12 = hydrido, perhaloaryloxy, alkanoylalkyl, alkanoylalkoxy, alkanoyloxy, N-aryl-N-alkylamino, heterocyclylalkoxy, etc.; with provisions] were prepd. for the treatment of atherosclerosis and other coronary artery diseases. I are useful as inhibitors of cholesteryl ester transfer protein (CETP; plasma lipid transfer protein-I). Examples include over 700 syntheses and data from two bioassays on CETP activity. For instance, reaction of 3-bromoaniline with 3-(1,1,2,2tetrafluoroethoxy)benzaldehyde in the presence of NaBH(OAc)3 and AcOH formed the secondary amine (96%). Addn. of 1,1,1-trifluoro-2,3epoxypropane in CH2Cl2 and Yb(OTf)3 gave the alc. (99%), which was silylated with tert-butyldimethylsilyl trifluoromethanesulfonate (58%). Heating a soln. of the tertiary amine with 4-chloro-3-ethylphenol, Cs2CO3, copper triflate benzene complex, and 1-naphthoic acid in 2:1 toluene:dimethylacetamide for 96 h gave II (23%). The latter inhibited CETP activity with IC50 values of 0.034 .mu.M and 0.88 .mu.M, resp., in the reconstituted buffer and human plasma assays.

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L25
    ANSWER 6 OF 8 CAPLUS COPYRIGHT 2003 ACS on STN
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2001:935384 CAPLUS AN

DN 136:69803

ΤI Preparation of N-benzothiazol-2-yl amides having affinity toward the A2A adenosine receptor

IN Alanine, Alexander; Flohr, Alexander; Miller, Aubry Kern; Norcross, Roger David; Riemer, Claus

F. Hoffmann-La Roche A.-G., Switz. PA

SO PCT Int. Appl., 160 pp. CODEN: PIXXD2

DT Patent

English LΑ

FAN.CNT 1

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PATENT NO.
                               KIND DATE
                                                              APPLICATION NO.
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PΙ
       WO 2001097786
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                                        20011227
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       WO 2001097786
                                A3
                                        20021212
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MAE	DAT 126.60002						

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IT 98-80-6, Phenylboronic acid 1692-15-5, 4-Pyridylboronic
 acid 1692-25-7, 3-Pyridylboronic acid 3900-89-8,
 2-Chlorophenylboronic acid 13331-27-6, 3-Nitrophenylboronic acid
 178752-79-9, 3-Dimethylaminophenylboronic acid
 RL: RCT (Reactant); RACT (Reactant or reagent)
 (prepn. of N-benzothiazolyl amides having affinity toward A2A adenosine receptor)

RN 98-80-6 CAPLUS

CN Boronic acid, phenyl- (9CI) (CA INDEX NAME)

RN 1692-15-5 CAPLUS CN Boronic acid, 4-pyridinyl- (9CI) (CA INDEX NAME)

RN 1692-25-7 CAPLUS

CN Boronic acid, 3-pyridinyl- (9CI) (CA INDEX NAME)

RN 3900-89-8 CAPLUS

CN Boronic acid, (2-chlorophenyl) - (9CI) (CA INDEX NAME)

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RN 13331-27-6 CAPLUS

CN Boronic acid, (3-nitrophenyl) - (9CI) (CA INDEX NAME)

RN 178752-79-9 CAPLUS

CN Boronic acid, [3-(dimethylamino)phenyl] - (9CI) (CA INDEX NAME)

GI

AB

The title compds. [I; R1 = H, alkyl, alkoxy, etc.; R2, R3 = H, halo, alkyl, alkoxy; R4 = H, alkyl, alkenyl, etc.; R = (un)substituted Ph, (CH2)n(5-6 membered (non)arom. heterocyclyl, (CH2)n+1Ph, etc.; n = 0-4; X = O, S, H2)], useful for the treatment of diseases related to the adenosine receptor, were prepd. Thus, reacting 2-amino-4-methoxy-7-phenylbenzothiazole with benzoyl chloride in pyridine afforded 69% I [R1 = OMe; R2, R3 = H; R4 = Ph; R = Ph; X = O]. Biol. data for compds. I were given.

L25 ANSWER 7 OF 8 CAPLUS COPYRIGHT 2003 ACS on STN

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- 131:271803 DN
- ΤI Thienyl-, furyl- and pyrrolyl-sulfonamides and derivatives thereof that modulate the activity of endothelin
- Chan, Ming Fai; Wu, Chengde; Raju, Bore Gowda; Kogan, Timothy; Kois, Adam; Verner, Erik Joel; Castillo, Rosario Silvestre; Yalamorri, IN Venkatachalapathi; Balaji, Vitukudi Narayanaiyengar Texas Biotechnology Corp., USA
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AU	726595	B2	20001116			
				ΑU	1996-55367	A 19960404
AU	762258	B2	20030619	ΑU	2001-57834	20010806
				ΑU	1998-61771	A319980220
US	2002018816	A1	20020214	US	2001-932245	20010817
US	6514518	B2	20030204			
			•	US	1993-138345	A319931018
				US	1995-464593	A219950605
				US	1998-27290	A119980220
US	2002091272	A1	20020711	US	2001-11610	20011105
			·	WO	1996-US4759	W 19960404
				US	1996-721183	A219960927
				US	1997-938325	A319970926

OS MARPAT 131:271803

IT 10365-98-7P, 3-Methoxyphenylboronic acid 16152-51-5P,
4-Isopropylphenylboronic acid 63139-21-9P, 4-Ethylphenylboronic
acid 134150-01-9P, 4-Propylphenylboronic acid
RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT

RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)

(intermediate; prepn. of thienyl-, furyl- and pyrrolyl-based sulfonamides and analogs as endothelin agonists and antagonists)

RN 10365-98-7 CAPLUS

CN Boronic acid, (3-methoxyphenyl) - (9CI) (CA INDEX NAME)

RN 16152-51-5 CAPLUS

CN Boronic acid, [4-(1-methylethyl)phenyl]- (9CI) (CA INDEX NAME)

RN 63139-21-9 CAPLUS

CN Boronic acid, (4-ethylphenyl) - (9CI) (CA INDEX NAME)

RN 134150-01-9 CAPLUS

CN Boronic acid, (4-propylphenyl) - (9CI) (CA INDEX NAME)

IT 98-80-6, Phenylboronic acid 5720-05-8, 4-Methylphenylboronic acid 5720-06-9, 2-Methoxyphenylboronic acid 5720-07-0, 4-Methoxyphenylboronic acid 13331-27-6 , 3-Nitrobenzeneboronic acid 13922-41-3, 1-Naphthaleneboronic acid 16419-60-6, 2-Methylphenylboronic acid 17933-03-8 , 3-Methylphenylboronic acid 30418-59-8, 3-Aminobenzeneboronic acid 40138-16-7, 2-Formylbenzeneboronic acid 73852-19-4 , 3,5-Bis(trifluoromethyl)benzeneboronic acid 94839-07-3, 3,4-(Methylenedioxy) phenylboronic acid 128796-39-4, 4-(Trifluoromethyl)benzeneboronic acid RL: RCT (Reactant); RACT (Reactant or reagent) (starting material; prepn. of thienyl-, furyl- and pyrrolyl-based sulfonamides and analogs as endothelin agonists and antagonists) 98-80-6 CAPLUS RN CN Boronic acid, phenyl- (9CI) (CA INDEX NAME)

RN 5720-05-8 CAPLUS

CN Boronic acid, (4-methylphenyl) - (9CI) (CA INDEX NAME)

RN 5720-06-9 CAPLUS

CN Boronic acid, (2-methoxyphenyl) - (9CI) (CA INDEX NAME)

RN 5720-07-0 CAPLUS

CN Boronic acid, (4-methoxyphenyl) - (9CI) (CA INDEX NAME)

RN 13331-27-6 CAPLUS

CN Boronic acid, (3-nitrophenyl) - (9CI) (CA INDEX NAME)

RN 13922-41-3 CAPLUS

CN Boronic acid, 1-naphthalenyl- (9CI) (CA INDEX NAME)

RN 16419-60-6 CAPLUS

CN Boronic acid, (2-methylphenyl) - (9CI) (CA INDEX NAME)

RN 17933-03-8 CAPLUS

CN Boronic acid, (3-methylphenyl) - (9CI) (CA INDEX NAME)

RN 30418-59-8 CAPLUS

CN Boronic acid, (3-aminophenyl) - (9CI) (CA INDEX NAME)

RN 40138-16-7 CAPLUS

CN Boronic acid, (2-formylphenyl) - (9CI) (CA INDEX NAME)

RN 73852-19-4 CAPLUS

CN Boronic acid, [3,5-bis(trifluoromethyl)phenyl] - (9CI) (CA INDEX NAME)

RN 94839-07-3 CAPLUS

CN Boronic acid, 1,3-benzodioxol-5-yl- (9CI) (CA INDEX NAME)

RN 128796-39-4 CAPLUS

CN Boronic acid, [4-(trifluoromethyl)phenyl]- (9CI) (CA INDEX NAME)

GI

AB Thienyl-, furyl- and pyrrolyl-sulfonamides, and methods for modulating or altering the activity of the endothelin family of peptides, are provided. In particular, the disclosure includes N-(isoxazolyl)thienylsulfonamides, N-(isoxazolyl) furylsulfonamides, and N-(isoxazolyl) pyrrolylsulfonamides, and methods using these sulfonamides for inhibiting the binding of an endothelin peptide to an endothelin receptor. The compds. are described by the formula Ar2SO2NHAr1 [I; Ar1 = (un) substituted aryl, particularly isoxazolyl; Ar2 = biol. effective group for inhibiting endothelin binding by .gtoreq. 50% at .ltoreq.100 .mu.M, notably thienyl, furyl, pyrrolyl, etc.]. Methods for treating endothelin-mediated disorders by administering effective amts. of I or their prodrugs are also provided. Such disorders include hypertension, cardiovascular disease, asthma, hypertension, inflammatory disease, glaucoma, etc. Approx. 190 synthetic examples are given, and numerous example compds. were prepd., tested, and/or claimed. For instance, 5-amino-4-bromo-3-methylisoxazole was treated with NaH in THF, followed by thiophene-2-sulfonyl chloride, to give 34% title compd. II. The similarly prepd. title compd. III had IC50 values of 0.024 .mu.M for ETA receptors and 7.95 .mu.M for ETB receptors, indicating substantial selectivity for ETA.

RE.CNT 64 THERE ARE 64 CITED REFERENCES AVAILABLE FOR THIS RECORD ALL CITATIONS AVAILABLE IN THE RE FORMAT

L25 ANSWER 8 OF 8 CAPLUS COPYRIGHT 2003 ACS on STN

Patel

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AN
     1999:262171 CAPLUS
     130:311785
DN
ΤI
     Preparation of heterocyclic benzenemethanamine derivatives as Ih ion
     channel modulators for use in psychotherapeutics
     Dijcks, Fredericus Antonius; Grove, Simon James Anthony; Carlyle, Ian
IN
     Craig; Thorn, Simon Nicholas; Rae, Duncan Robertson; Ruigt, Gerardus
     Stephanus Franciscus; Leysen, Dirk
PΑ
     Akzo Nobel N.V., Neth.
SO
     PCT Int. Appl., 50 pp.
     CODEN: PIXXD2
DT
     Patent
     English
LΑ
FAN.CNT 1
     PATENT NO.
                      KIND DATE
                                           APPLICATION NO.
                                                            DATE
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PΙ
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                      A2 · 19990422
                                           WO 1998-EP6651
                                                            19981014
     WO 9918941
                     A3 20000113
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             RU, SG, SI, SK, SL, TR, TT, UA, US, UZ, VN, YU, AM, AZ, BY, KG,
             KZ, MD, RU, TJ, TM
         RW: GH, GM, KE, LS, MW, SD, SZ, UG, ZW, AT, BE, CH, CY, DE, DK, ES,
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                                           US 1997-950359 A 19971014
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                                                           19981014
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             IE, FI
                                           US 1997-950359 A 19971014
                                           WO 1998-EP6651 W 19981014
     JP 2003510242
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                            20030318
                                           JP 2000-515576
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                                           WO 1998-EP6651 W 19981014
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     US 6313139
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                                                            19990722
                                           US 1997-950359 A319971014
                                           US 2001-933192
     US 2002037885
                       Α1
                            20020328
                                                            20010820
                                           US 1999-359284 A319990722
OS
     MARPAT 130:311785
ΙT
     40138-16-7, 2-Formylbenzeneboronic acid
     RL: RCT (Reactant); RACT (Reactant or reagent)
        (starting material; prepn. of heterocyclic benzenemethanamine derivs.
        as Ih channel modulators for psychotherapeutics)
     40138-16-7 CAPLUS
RN
     Boronic acid, (2-formylphenyl) - (9CI) (CA INDEX NAME)
CN
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Page 88

GI

$$CH_2$$
 II CH_2 III

AΒ The invention relates to the use of Ih channel modulators, which modulate the hyperpolarization-activated cation current (Ih), in the manuf. of medicaments for use in psychiatry. In particular, it relates to certain novel methanamine derivs. A-B-(CH2)nCH(NH2)CH2C(R3):CR4R5 [I; A = certain (un) substituted and optionally benzo- or pyrido-fused arom. 5- or 6-membered hetero- and carbocycles, e.g., isoxazol-3-yl, or (un) substituted cyclohexylmethyl; B = (un) substituted Ph, furyl, thienyl, benzofuryl, benzothienyl, or naphthyl; n = 0 or 1; R3, R4, R5 = halo, alkyl, or H; or R3R4 = bond], to processes for their prepn., to pharmaceutical formulations contg. them, and to their use in medical therapy, particularly as antidepressants, anxiolytics, and antipsychotics. Examples include prepns. of over 50 compds. I and numerous intermediates, and biol. data for several prepd. and known compds. For instance, 2-(benzo[b] furan-2-yl) benzaldehyde (prepn. given) was treated with LiN(SiMe3)2 in THF and then with allylmagnesium bromide to give, after workup and acidification, title compd. II.HCl. The similarly prepd. title compd. III (as the maleate) inhibited marble-burying behavior in mice with an ED50 of 1.7 mg/kg s.c. This in vivo effect by I correlated with Ih channel inhibition in vitro.

=> log y		
COST IN U.S. DOLLARS	SINCE FILE	TOTAL
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                 Data from 1960-1976 added to RDISCLOSURE
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         Jul 21
                 Identification of STN records implemented
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         Jul 21
                 Polymer class term count added to REGISTRY
NEWS
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      7
         Jul 22
                 INPADOC: Basic index (/BI) enhanced; Simultaneous Left and
NEWS
                 Right Truncation available
NEWS
      8
         AUG 05
                 New pricing for EUROPATFULL and PCTFULL effective
                 August 1, 2003
NEWS
     9
         AUG 13
                 Field Availability (/FA) field enhanced in BEILSTEIN
                 PATDPAFULL: one FREE connect hour, per account, in
NEWS 10
         AUG 15
                 September 2003
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NEWS 11
                 September 2003
NEWS 12
         AUG 15
                 RDISCLOSURE: one FREE connect hour, per account, in
                 September 2003
NEWS 13
         AUG 15
                 TEMA: one FREE connect hour, per account, in
                 September 2003
NEWS 14
         AUG 18
                 Data available for download as a PDF in RDISCLOSURE
NEWS 15
         AUG 18
                 Simultaneous left and right truncation added to PASCAL
         AUG 18
                 FROSTI and KOSMET enhanced with Simultaneous Left and Righ
NEWS 16
                 Truncation
         AUG 18
                 Simultaneous left and right truncation added to ANABSTR
NEWS 17
NEWS 18
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              April 4 CURRENT WINDOWS VERSION IS V6.01a, CURRENT
NEWS EXPRESS
              MACINTOSH VERSION IS V6.0b(ENG) AND V6.0Jb(JP),
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10085368.23 Page 2

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L1 STRUCTURE UPLOADED

=> d l1 L1 HAS NO ANSWERS L1 STR

$$\begin{bmatrix} J_{0-1} & G_1 \\ J_{0-1} & G_1 \\ \end{bmatrix}$$

G1 C,O,S,N,CH,NH

Structure attributes must be viewed using STN Express query preparation.

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100.0% PROCESSED 19899 ITERATIONS

210 ANSWERS

SEARCH TIME: 00.00.01

L2 210 SEA SSS FUL L1

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FULL ESTIMATED COST

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FILE COVERS 1907 - 24 Sep 2003 VOL 139 ISS 13

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Page 4

FILE LAST UPDATED: 23 Sep 2003 (20030923/ED)

This file contains CAS Registry Numbers for easy and accurate substance identification.

=> s 12

309 L2 L3

=> s 13 and syn thesis and preparation

0 L3 AND SYN THESIS AND PREPARATION L4

=> s 13 and synthesis

48 L3 AND SYNTHESIS

=> s 15 and lithium

3 L5 AND LITHIUM L6

=> d 16 fbib hitstr abs total

ANSWER 1 OF 3 CAPLUS COPYRIGHT 2003 ACS on STN L6

1997:381760 CAPLUS \cdot AN

127:121799 DN

Synthesis and structural characterization of some novel ΤI metalloboroxides bearing boron-bound mesityl and fluoromesityl substituents: the molecular structure of the first metallaboroxane complex

ΑU Gibson, Vernon C.; Redshaw, Carl; Clegg, William; Elsegood, Mark R. J.

Dep. Chem., Imperial Coll., South Kensington, London, SW7 2AY, UK Polyhedron (1997), 16(15), 2637-2641 CS

SO CODEN: PLYHDE; ISSN: 0277-5387

PB Elsevier

Journal DT

English LΑ

IT192823-38-4P

> RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)

(prepn. and metalation reaction with lithium and molybdenum)

RN 192823-38-4 CAPLUS

Borinic acid, bis[2,4,6-tris(trifluoromethyl)phenyl]- (9CI) (CA INDEX CN NAME)

IT 192823-43-1

> RL: RCT (Reactant); RACT (Reactant or reagent) (reaction with copper dibromide)

RN192823-43-1 CAPLUS

CN Borinic acid, bis[2,4,6-tris(trifluoromethyl)phenyl]-, lithium salt (9CI) (CA INDEX NAME)

● Li

Treatment of the new boronous acid HOB(fmes)2 (1) (fmes = 2,4,6-(CF3)3C6H2) with BuLi in Et2O/pentane affords, after work-up, the dimer [Li(THF)OB(fmes)2]2 (2). Reaction of [Mo2(NMe2)6] with two equiv. of 1 in toluene gives the amido-boroxide complex Mo2(NMe2)4[OB(fmes)2]2 (3). Treatment of CuBr2 with LiOB(mes)2 (mes = 2,4,6-Me3C6H2) in THF affords after work-up and addn. of excess pyridine the monomeric Cu(II) boroxide, {Cu[O3B2(mes)2]2[Li(MeCN)(C5H5N)]2} (4), contg. the new ligand mesB(O)OB(O)mes, as a result of loss of mesitylene and formation of a B-O-B bond. 2 And 4 were structurally characterized by x-ray crystallog.

L6 ANSWER 2 OF 3 CAPLUS COPYRIGHT 2003 ACS on STN

AN 1987:469592 CAPLUS

DN 107:69592

TI **Synthesis** and spectroscopic and structural characterization of derivatives of the quasi-alkoxide ligand [OBMes2] - (Mes = 2,4,6-Me3C6H2)

AU Weese, Kenneth J.; Bartlett, Ruth A.; Murray, Brendan D.; Olmstead, Marilyn M.; Power, Philip R.

CS Dep. Chem., Univ. California, Davis, CA, 95616, USA

SO Inorganic Chemistry (1987), 26(15), 2409-13

CODEN: INOCAJ; ISSN: 0020-1669

DT Journal

LA English

IT 20631-84-9

RL: PRP (Properties)
(crystal structure of)

RN 20631-84-9 CAPLUS

CN Borinic acid, bis(2,4,6-trimethylphenyl) - (9CI) (CA INDEX NAME)

AB Treatment of Mes2BOH; (Mes = 2,4,6-Me3C6H2) with BuLi in hexane/ether affords a suspension of LiOBMes2, which can be crystd. from THF soln. as the dimer [{Li(THF)OBMes2}2] (II). Treatment of a slurry of anhyd. CoCl2 in THF with 2 equiv. of II gives [Co{OBMes2}2Li(THF)2Cl2Li(THF)2] (III) in good yield. The x-ray crystal structures of I, II, and III are also reported. The structure of I is the 1st for a diorganoboronous acid, and it exists in the solid state as H-bonded tetramers. II is the 1st structurally characterized example of a metal salt of a boronous acid, and it possesses a dimeric structure previously seen only with very bulky

-OC(tert-Bu)3 and -OC6H2-2,6-tert-Bu2-4-Me salts. III has Co pseudotetrahedrally bound to 2 OBMes2 and Cl- ligands, which also form bridges to 2 Li+ ions. Each Li+ is also pseudotetrahedrally coordinated, with 2 THF donors as the remaining ligands in each case.

ANSWER 3 OF 3 CAPLUS COPYRIGHT 2003 ACS on STN L6 ΑN 1960:44693 CAPLUS 54:44693 DN OREF 54:8842f-i,8843a-c Synthesis and structure of aromatic boron compounds ΤI Davidson, J. M.; French, C. M. ΑU CS Queen Mary Coll., London SO Journal of the Chemical Society, Abstracts (1960) 191-5 CODEN: JCSAAZ; ISSN: 0590-9791 DT Journal LΑ Unavailable IT 2622-89-1, Borinic acid, diphenyl- 131732-34-8, Borinic acid, 2-biphenylylphenyl-(prepn. of) 2622-89-1 CAPLUS RN Borinic acid, diphenyl- (6CI, 7CI, 8CI, 9CI) (CA INDEX NAME)

CN

131732-34-8 CAPLUS RNBorinic acid, 2-biphenylylphenyl- (6CI) (CA INDEX NAME) CN

10-Hydroxy-9-oxa-10-boraanthracene (I) was prepd. and its aromatic AB character demonstrated by ultraviolet spectroscopy. The mechanism of the reaction of Bu metaborate (II) with Grignard and Li reagents was investigated and the conditions under which org. boronous or boronic acid was the predominant product were examd. An attempt to prep. 9-diethylamino-9-borafluorene was also described. PhMgBr (from 15.7 g. PhBr) in 50 ml. Et20 treated dropwise under reflux with 10 g. phenylboronic anhydride (III) in 75 ml. C6H6, refluxed a further 0.5 hr., the mixt. hydrolyzed with 200 ml. 15% HCl, the solvents removed, 20 ml. ligroine added, and the mixt. filtered gave 3.5 g. III, m. 214.degree... Removal of the solvent from the filtrate gave 11.5 g. diphenylboronous acid (IV), n20D 1.5907. IV with HOCH2CH2NH2 formed 65% 2-aminoethyl diphenylboronite, m. 187.degree.. 2-Biphenylylphenylboronous acid (V) was similarly prepd. front 7.5 g. III and 1 mole 2-biphenylylmagnesium iodide in Et20, after hydrolysis, ethanolamine added, and crystd. to give 10.2 q. 2-aminoethyl-2-biphenylyl phenylboronite (VI), m. 175.degree. (alc.). VI (3 g.) shaken with 30 ml. Et20 and 30 ml. 10% HCl gave 2.55 g. V, viscous liquid. Mg (0.7 g.) reacted readily with 7.5 g. 2-iododiphenyl ether and

3 g. II in 60 ml. Et20 after addn. of iodine; after 10 min. of spontaneous refluxing and 0.5 hr. of heating the mixt. was hydrolyzed with 100 ml. 15% HCl, the acid products extd. with 5% NaOH, and the basic ext. acidified to give 1.5 g. o-phenoxyphenylboronic acid, m. 114.degree. (C6H6-cyclohexane). 9-Bromophenanthrene (5 g.) and 2.5 g. II gave 2.45 g. 9-phenanthrylboronic acid, m. 324.degree. (H2O). 2,2'-Dilithiodiphenyl ether in 156 ml. Et2O treated during 10 min. with 6.7 g. II in 25 ml. Et20, the soln. refluxed 2 hrs., and hydrolyzed with 100 ml. 10% HCl gave 5.9 g. 10-hydroxy-9-oxa-10-boraanthracene (VII), m. 285.degree. (C6H6-cyclohexane). The same soln. of 2,2'-dilithiodiphenyl ether (600 ml.) and 200 ml. ether soln. contg. 37 g. BF3-Et20 simultaneously added to 100 ml. Et20 under N during 45 min. and the mixt. refluxed 1 hr. gave 11.1 g. VII. 2-Biphenylylmagnesium iodide (from 10 g. 2-iodobiphenyl) in 50 ml. Et20 treated rapidly with 3.5 g. II in 15 ml. Et20, and the soln. refluxed 0.5 hr. gave 5 g. 2-biphenylylboronic acid (VIII), m. 121-3.degree. (H2O), resolidified to the anhydride, m. 195.degree.. VIII (3.9 q.) esterified with alc. by azeotropic distn. gave 3.4 q. di-Et ester, b4 136-8.degree., n20D 1.5444. Di-Bu 2-biphenylylboronate (IX) was prepd. by direct esterification of the Grignard reaction mixt. after hydrolysis. 2-Iodobiphenyl (26.5 g.) and 8.8 g. II afforded 13.3 g. IX, b0.6 149-51.degree., n20D 1.5310. IX (7.5 g.) heated 18 hrs. at 140.degree. with 11 g. PCl5 gave 4.35 g. 2-biphenylylboron dichloride, b0.25 95-6.degree., n20D 1.5661. 2,2'-Dilithiobiphenyl (from 4 g. 2,2'-diiodobiphenyl) in 60 ml. Et2O slowly treated with 0.9 q. II in 15 ml. Et20 under N, the soln. refluxed 15 min., hydrolyzed with dil. NH4Cl, and the soln. azeotropically distd. with HOCH2CH2NH2 and PhMe gave 1.3 q. bis(2-aminoethyl)2-biphenylyl boronate, m. 134.degree. (C6H6). A sample was hydrolyzed with dil. HCl to the acid which was dried to form the anhydride, m. 206.degree. (cyclohexane).

=> d his

L3

L5

(FILE 'HOME' ENTERED AT 16:14:11 ON 24 SEP 2003)

FILE 'REGISTRY' ENTERED AT 16:14:32 ON 24 SEP 2003 STRUCTURE UPLOADED

L1 STRUCTURE UPL L2 210 S L1 SSS FULL

FILE 'CAPLUS' ENTERED AT 16:15:16 ON 24 SEP 2003

309 S L2

L4 0 S L3 AND SYN THESIS AND PREPARATION

48,S L3 AND SYNTHESIS

L6 3 S L5 AND LITHIUM

=> s 15 and magnesium

L7 2 L5 AND MAGNESIUM

=> d 17 fbib hitstr abs total

L7 ANSWER 1 OF 2 CAPLUS COPYRIGHT 2003 ACS on STN

AN 2003:150618 CAPLUS

DN 138:190256

TI Process for the preparation or aryl and alkyl boron compounds in micro reactors

IN Koch, Manfred; Wehle, Detlef; Scherer, Stefan; Forstinger, Klaus; Meudt, Andreas; Hessel, Volker; Werner, Bernd; Loewe, Holger

PA Clariant Gmbh, Germany

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SO
     Eur. Pat. Appl., 12 pp.
     CODEN: EPXXDW
DT
     Patent
LΑ
    German
FAN.CNT 1
     PATENT NO.
                      KIND DATE
                                           APPLICATION NO. DATE
                            20030226
                                           EP 2002-16149
                                                            20020720
PΙ
    EP 1285924
                      A1
         R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT,
             IE, SI, LT, LV, FI, RO, MK, CY, AL, TR, BG, CZ, EE, SK
                                           DE 2001-10140857A 20010821
     DE 10140857
                       Α1
                            20030306
                                           DE 2001-10140857 20010821
                                                           20020801
                            20030529
    US 2003100792
                       A1
                                           US 2002-210807
                                           DE 2001-10140857A 20010821
                                           JP 2002-240103
    JP 2003128677
                       Α2
                            20030508
                                                           20020821
                                           DE 2001-10140857A 20010821
     2622-89-1P, Diphenylborinic acid
IT
     RL: SPN (Synthetic preparation); PREP (Preparation)
        (synthesis of trialkyl- and triaryl-substituted boranes,
       boronic acids, and tetraalkylborates in flow-through reactors))
     2622-89-1 CAPLUS
RN
     Borinic acid, diphenyl- (6CI, 7CI, 8CI, 9CI) (CA INDEX NAME)
CN
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Ph | Ph— B— OH

PΙ

Patel

JP 06345756

AB Manuf. of arylboron and alkylboron compds., of general formulas RnBX3-n and R4B-MgY+, as well as RnB(OH)3-n (prepd. by hydrolysis of RnBX3-n), are prepd. from the corresponding arylmagnesium halides and alkylmagnesium halides, R-Mg-Y, and BX3, in which X = F, Cl, Br, I, Cl-5-alkoxy, N,N-di(Cl-5-alkyl)amino, or (Cl-5-alkyl)thio; n = 1, 2, or 3; and R = Cl-6-alkyl, (RO-, RR'N-, Ph-, substituted Ph-, F-, and RS-), and (Cl-6-alkyl)-substituted phenyl; and (Cl-6-alkyl-, Cl-6-alkoxy-, Cl-5-thioalkyl-, silyl- F-, Cl-, dialkylamino-, diarylamino-, and alkylarylamino)-substituted Ph, in addn. to heterocycloaryl substituents with one or two heteroatoms (e.g., N, O, or S). The compds. are synthesized in through-flow microreactors in flow channels of diam. 0.25 .mu. to 1.5 mm.

RE.CNT 3 THERE ARE 3 CITED REFERENCES AVAILABLE FOR THIS RECORD ALL CITATIONS AVAILABLE IN THE RE FORMAT

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L7
     ANSWER 2 OF 2 CAPLUS COPYRIGHT 2003 ACS on STN
     1995:444247 CAPLUS
ΑN
DN
     122:213862
TI
     Preparation of sesamol
     Kumamoto, Nobumitsu; Uchibori, Yukitaka; Umeno, Masayuki
IN
     Hokko Chem Ind Co, Japan
PA
SO
     Jpn. Kokai Tokkyo Koho, 5 pp.
     CODEN: JKXXAF
DT
     Patent
LΑ
     Japanese
FAN.CNT 1
     PATENT NO.
                      KIND
                            DATE
                                           APPLICATION NO.
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19941220

A2

9/24/2003>

JP 1993-156387

19930603

10085368.23

Page 9

JP 2614812

B2 19970528

JP 1993-156387 19930603

OS CASREACT 122:213862; MARPAT 122:213862

IT 161800-66-4P

RN 161800-66-4 CAPLUS

CN Borinic acid, bis(1,3-benzodioxol-5-yl)- (9CI) (CA INDEX NAME)

GI

AB Sesamol (I) is prepd. in several steps from benzenemagnesium halide II [X = halo]. Thus, reaction of II [X = Br] with tri-Bu borate, followed by treatment with aq. 10% sulfuric acid soln., and reaction with H2O2, gave 80.2% I.

=> s grignard reaction and lithium and magnesium
L8 194 GRIGNARD REACTION AND LITHIUM AND MAGNESIUM

=> s 18 and phenyl

L9 70 L8 AND PHENYL

=> s 18 and phenyl and chloride

L10 30 L8 AND PHENYL AND CHLORIDE

=> s 18 and pyridine

L11 10 L8 AND PYRIDINE

=> s 18 and pyrimidine

L12 1 L8 AND PYRIMIDINE

. => s 18 and pyridazine

L13 0 L8 AND PYRIDAZINE

=> s 18 and furan

L14 2 L8 AND FURAN

=> s 18 and thien

L15 1 L8 AND THIEN

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=> d his
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(FILE 'HOME' ENTERED AT 16:14:11 ON 24 SEP 2003)

FILE 'REGISTRY' ENTERED AT 16:14:32 ON 24 SEP 2003

L1 STRUCTURE UPLOADED

L2 210 S L1 SSS FULL

FILE 'CAPLUS' ENTERED AT 16:15:16 ON 24 SEP 2003

L3 309 S L2

L4 0 S L3 AND SYN THESIS AND PREPARATION

L8 194 S GRIGNARD REACTION AND LITHIUM AND MAGNESIUM

L9 70 S L8 AND PHENYL

L10 30 S L8 AND PHENYL AND CHLORIDE

L11 10 S L8 AND PYRIDINE
L12 1 S L8 AND PYRIMIDINE
L13 0 S L8 AND PYRIMIDINE
L14 2 S L8 AND FURAN
L15 1 S L8 AND THIEN

=> s 18 and chloride

L16 84 L8 AND CHLORIDE

=> s 116 and phenyl and pyridine and furan and thien and pyrimidine L17 0 L16 AND PHENYL AND PYRIDINE AND FURAN AND THIEN AND PYRIMIDINE

=> s 18 and pyridine and chliride

L18 0 L8 AND PYRIDINE AND CHLIRIDE

=> s 18 and pyrimidine and chloride

L19 1 L8 AND PYRIMIDINE AND CHLORIDE

=> s 18 and furan and chloride

L20 1 L8 AND FURAN AND CHLORIDE

=> s 18 and thien and chloride

L21 1 L8 AND THIEN AND CHLORIDE

=> d l16 fbib hitstr abs total

L16 ANSWER 1 OF 84 CAPLUS COPYRIGHT 2003 ACS on STN

AN 2003:656756 CAPLUS

DN 139:197491

TI Process for preparing optically active azole derivatives

IN Suzuki, Tsuneji; Tsunoda, Hidetoshi

PA Mitsui Chemicals, Inc., Japan

SO PCT Int. Appl., 53 pp.

CODEN: PIXXD2

DT Patent

LA Japanese

FAN.CNT 1

PATENT NO. KIND DATE APPLICATION NO. DATE
PI WO 2003068758 A1 20030821 WO 2003-JP1308 20030207

W: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NO, NZ, OM, PH, PL, PT, RO, RU, SC, SD, SE, SG, SK, SL, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, YU, ZA, ZM, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM
RW: GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZM, ZW, AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IT, LU, MC, NL, PT, SE, SI, SK, TR, BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG

JP 2002-37966 A 20020215 JP 2002-236368 A 20020814

GI

AB The title compds. I [R1 = (un)substituted alkyl, etc.; R5, R6 = halo, etc.; the asterisk indicates asym. carbon], useful as pharmaceutical and agrochem. intermediates, are prepd. in a multistep process involving a highly diastereoselective arylation reaction of an optically active azole alkyl ketone deriv. which is prepd. from triazolylacetic acid (or salt thereof) and an optically active deriv. of 2-hydroxypropionic acid ester. Thus, (2S,3R)-2-(2,4-difluorophenyl)-1-(1H-1,2,4-triazol-1-yl)-2,3-butanediol was prepd. by the title process.

RE.CNT 7 THERE ARE 7 CITED REFERENCES AVAILABLE FOR THIS RECORD
ALL CITATIONS AVAILABLE IN THE RE FORMAT

L16 ANSWER 2 OF 84 CAPLUS COPYRIGHT 2003 ACS on STN

AN 2002:882089 CAPLUS

DN 137:384754

TI Preparation of exo-biperidens via the coupling of exo-silylenolethers with N-methylenepiperidinium salts

IN Grosse, Markus; Klein, Peter; Thyes, Marco; Weber, Klaus Martin;
Vilsmaier, Elmar

PA Abbott Gmbh & Co. KG, Germany

SO Ger. Offen., 12 pp.

CODEN: GWXXBX

DT Patent

LA German

FAN.CNT 1

PATENT NO. KIND DATE APPLICATION NO. DATE
PI DE 10124453 A1 20021121 DE 2001-10124453 20010518

9/24/2003>

Patel

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WO 2002096874
                                20021205
                                                     WO 2002-EP5497
                                                                             20020517
                         A2
WO 2002096874
                        A3
                                20030130
          AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN,
           CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, ES, FI, GB, GD, GE, GH,
           GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR,
           LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NO, NZ, OM, PH,
           PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VN, YU, ZA, ZM, ZW, AM, AZ, BY, KG, KZ, MD, RU,
           TJ, TM
     RW: GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZM, ZW, AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, TR, BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG
                                                     DE 2001-10124453A 20010518
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OS CASREACT 137:384754; MARPAT 137:384754

GΙ

III

AB A process for the prepn. of exo-biperidens I via the coupling of exo-silylenolethers II [R = alkyl, cycloalkyl] with N-methylenepiperidinium salts is disclosed. For example, coupling of exo-silylenolether II (R = Me), e.g., prepd. from cyclopentadiene and 3-buten-2-one in 2-steps, and N-methylenepiperidinium chloride, followed by the addn. of chlorophenylmagesium afforded a diastereomeric mixt. of exo-biperidens I. A key step of the process is the NaOMe mediated isomerization of endo-1-(bicyclo[2.2.1]hept-5-en-2-yl)ethan-2-one to the exo isomer.

L16 ANSWER 3 OF 84 CAPLUS COPYRIGHT 2003 ACS on STN

AN 2002:590289 CAPLUS

DN 137:140630

TI Preparation of substituted silanes via organometallic catalysts mediated reaction of alkyl magnesium compounds with halosilane

IN Sims, Philip Franklin; Schwindeman, James Anthony

PA FMC Corporation, USA

SO U.S., 6 pp. CODEN: USXXAM

DT Patent LA English

FAN. CNT 1

OS CASREACT 137:140630; MARPAT 137:140630

AB Processes for the synthesis of substituted silanes from alkyl magnesium compds. using a mixt. of catalysts. The catalyst systems include both a copper halide and a salt of a Group IA, IIA, or IVA element or a transition metal. Thus, CuCl/KCN catalyzed reaction of t-butylmagnesium chloride with Me2SiCl2 in THF gave 91% t-butylchlorodimethylsilane.

RE.CNT 26 THERE ARE 26 CITED REFERENCES AVAILABLE FOR THIS RECORD ALL CITATIONS AVAILABLE IN THE RE FORMAT

L16 ANSWER 4 OF 84 CAPLUS COPYRIGHT 2003 ACS on STN

AN 2001:880027 CAPLUS

DN 136:166979

TI Disparate Roles of Chiral Ligands and Molecularly Imprinted Cavities in Asymmetric Catalysis and Chiral Poisoning

AU Koh, Jeong Hwan; Larsen, Andrew O.; White, Peter S.; Gagne, Michel R.

CS Department of Chemistry, University of North Carolina, Chapel Hill, NC, 27599-3290, USA

SO Organometallics (2002), 21(1), 7-9 CODEN: ORGND7; ISSN: 0276-7333

Ι

PB American Chemical Society

DT Journal

LA English

GI

AB The activation of molecularly imprinted metal complexes generated Lewis acid catalysts, prepd. via copolymn. of metallomonomers (I; X = Cl, X2 = O,O-dideprotonated (S)-, (R)-BINOL; Ar = p-C6H4C(CH3):CH2) with EDMA (ethylene dimethacrylate), for the ene reaction, each of which contains a chiral diphosphine ligand and a chiral BINOL-shaped cavity. Poisoning expts. with (R)- and (S)-BINAM (where (R)- and (S)-BINAM = (R)- and (S)-1,l'-binaphthyl-2,2'-diamine, resp.) indicated that while the chiral cavity can differentiate the chiral poisons, it is the chiral diphosphine ligand which controls the enantioselectivity of the ene product.

RE.CNT 22 THERE ARE 22 CITED REFERENCES AVAILABLE FOR THIS RECORD ALL CITATIONS AVAILABLE IN THE RE FORMAT

Page 14

- L16 ANSWER 5 OF 84 CAPLUS COPYRIGHT 2003 ACS on STN
- AN 2001:662289 CAPLUS
- DN 135:371815
- TI Zirconium Phosphinimide Complexes: Synthesis, Structure, and Deactivation Pathways in Ethylene Polymerization Catalysis
- AU Yue, Nancy; Hollink, Emily; Guerin, Fred; Stephan, Douglas W.
- CS School of Physical Sciences Chemistry and Biochemistry, University of Windsor, Windsor, ON, N9B 3P4, Can.
- SO Organometallics (2001), 20(21), 4424-4433 CODEN: ORGND7; ISSN: 0276-7333
- PB American Chemical Society
- DT Journal
- LA English
- OS CASREACT 135:371815
- Zirconium phosphinimide complexes of the form CpZr(NP-t-Bu3)Cl2 (1) and AΒ Cp*Zr(NPR3)Cl2 (R = i-Pr (2), t-Bu (3)) were readily prepd. under ambient conditions via the reaction of [CpZrCl3]n or Cp*ZrCl3 with the appropriate trialkylphosphinimide lithium salt (R3PNLi). A series of derivs. were readily obtained via alkylation or arylation of the above dihalide precursors. These included CpZr(NP-t-Bu3)Me2 (4), Cp*Zr(NPR3)Me2 (R = i-Pr (5), t-Bu (6)), CpZr(NP-t-Bu3)Ph2 (7), Cp*Zr(NPR3)Ph2 (R = i-Pr(8), t-Bu (9)), CpZr(NP-t-Bu3)Bn2 (10), CpZr(NP-t-Bu3)(CH2SiMe3)2 (11), Cp*Zr(NPR3)(ally1)2(R = i-Pr(12), t-Bu(13)), Cp*Zr(NPR3)(Cp)Cl(R = i-Pr(12), t-Bu(13))i-Pr(14), t-Bu(15)), and Cp*Zr(NPR3)(CH2C(CH3)C(CH3)CH2) (R = i-Pr(16), t-Bu (17)). Reaction of 17 with the borane B(C6F5)3 yielded the zwitterionic and cationic complexes Cp*Zr(NP-t-Bu3)(CH2C(CH3)C(CH3)CH2B(C6F5)3) (18) and Cp*Zr(NP-t-Bu3)(THF)(CH2C(CH3)C(CH3)CH2B(C6F5)3) (19). A no. of the above compds. were screened for their potential as catalyst precursors in ethylene polymn. In general, upon activation with methylaluminoxane, the resulting catalysts exhibit low activity. Efforts to understand the deactivation pathway for these zirconium catalysts involved investigating the interactions of catalyst precursors with activators. For example, reaction of 4 with the borane B(C6F5)3 leads to aryl group transfer and formation of catalytically inactive CpZr(NP-t-Bu3)(C6F5)2 (20). Interactions with MAO were modeled via reaction with AlMe3. The Zr clusters (Cp*Zr)4(.mu.-Cl)5(Cl)(.mu.-CH)2 (21) and (Cp*Zr)5(.mu.-Cl)6(.mu.-CH)3 (22) were two of the products that were characterized from these reactions. The isolation of 21 and 22 infers that aryl for Me exchange, ligand abstraction, and C-H bond activation may be catalyst deactivation pathways.
- RE.CNT 33 THERE ARE 33 CITED REFERENCES AVAILABLE FOR THIS RECORD ALL CITATIONS AVAILABLE IN THE RE FORMAT
- L16 ANSWER 6 OF 84 CAPLUS COPYRIGHT 2003 ACS on STN
- AN 2001:321295 CAPLUS
- DN 135:92230
- TI A detailed ab initio MO investigation of the diastereoselectivities of five- and six-membered ring ketones bearing O and S, C and S, and C and O substituents at the .alpha.-carbon
- AU Yadav, V. K.; Sriramurthy, V.
- CS Department of Chemistry, Indian Institute of Technology, Kanpur, 208 016, India
- SO Tetrahedron (2001), 57(18), 3987-3995 CODEN: TETRAB; ISSN: 0040-4020

10085368.23 Page 15

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PΒ
     Elsevier Science Ltd.
DT
     Journal
LΑ
     English
     The steric effects in the geometry on cation-chelation predict the exptl.
AB
     .pi.-selectivity of 1-oxa-4-thiaspiro[4.5]decan-6-one,
     1-oxa-4-thiaspiro[4.4] nonan-6-one, 1-thiaspiro[4.4] nonan-6-one, and
     1-oxaspiro[4.4] nonan-6-one. The reversal in the selectivity of
     1-oxa-4-thiaspiro[4.4] nonan-6-one on redn. with (i-Bu)2AlH appears to be a
     direct consequence of the steric interactions arising from the large i-Bu
     substituents. The antiperiplanar effects are not as significant as the
     steric effects. An ab initio MO investigation of the
     diastereoselectivities five- and six-membered ring ketones bearing O and
     S, C and S, and C and O substituents with the application of the
     cation-complexation model is described.
RE.CNT 72
              THERE ARE 72 CITED REFERENCES AVAILABLE FOR THIS RECORD
             ALL CITATIONS AVAILABLE IN THE RE FORMAT
    ANSWER 7 OF 84 CAPLUS COPYRIGHT 2003 ACS on STN
     2000:493491 CAPLUS
AN
DN
     133:89250
     Method for producing substituted cyclopentadienes from the reaction of
ΤI
     organometallic compounds with substituted 2-cyclopentenones followed by
     hydrolysis and dehydration with strong mineral acids
     Bingel, Carsten
IN
     Targor G.m.b.H., Germany
PΑ
     PCT Int. Appl., 17 pp.
SO
     CODEN: PIXXD2
DT
     Patent
LΑ
     German
FAN.CNT 2
                      KIND DATE
                                          APPLICATION NO.
                                                            DATE
     PATENT NO.
                      _ _ _ _
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                                          WO 2000-EP15
     WO 2000041987
                      A1 20000720
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PΤ
         W: JP, US
         RW: AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL,
             PT, SE
                                           DE 1999-19900732A 19990112
                                           DE 1999-19935885A 19990730
     DE 19900732
                      A1
                            20000713
                                           DE 1999-19900732 19990112
     DE 19935885
                      A1
                            20010201
                                           DE 1999-19935885 19990730
                            20010228
                                           EP 2000-113883 20000630
     EP 1078905
                      A1
           AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT,
             IE, SI, LT, LV, FI, RO
                                           DE 1999-19935885A 19990730
     JP 2001064210
                       A2
                            20010313
                                           JP 2000-215576 20000717
                                           DE 1999-19935885A 19990730
PATENT FAMILY INFORMATION:
FAN 2000:475955
                                           APPLICATION NO. DATE
     PATENT NO.
                      KIND DATE
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     DE 19900732
                            20000713
                                           DE 1999-19900732 19990112
PΙ
                      A1
     WO 2000041987
                            20000720
                                           WO 2000-EP15
                                                            20000104
                      A1
         W: JP, US
         RW: AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL,
             PT, SE
                                           DE 1999-19900732A 19990112
                                           DE 1999-19935885A 19990730
     CASREACT 133:89250; MARPAT 133:89250
OS
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GI

$$R^{6}$$
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 R^{3}
 R^{2}
 R^{4}
 R^{5}
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 R^{1}
 R^{4}

AB In the title process, 3-substituted-2-cyclopenten-1-ones (I; R = C1-20 hydrocarbyl; R4-R6 = H, alkyl, aryl) (e.g., 3-methyl-2-cyclopenten-1-one) are reacted with organometallic compds. R2(R3)M [M = Li, MgCl, MgBr, MgI; R2, R3 = H, (un)branched alkyl, aryl] (e.g., n-BuLi) to form alkoxide intermediates (II) which are protonated, hydrolyzed, and dehydrated with aq. solns. of strong mineral acids (e.g., aq. sulfuric acid) to form the desired products (III; 1-butyl-3-methylcyclopentadiene) and byproducts (IV) which are both extd. into an org. phase (e.g., toluene) and sep. worked up.

RE.CNT 1 THERE ARE 1 CITED REFERENCES AVAILABLE FOR THIS RECORD ALL CITATIONS AVAILABLE IN THE RE FORMAT

L16 ANSWER 8 OF 84 CAPLUS COPYRIGHT 2003 ACS on STN

AN 2000:475955 CAPLUS

DN 133:89249

TI Procedure for the production of 1,3-disubstituted cyclopentadienes by the condensation of organometallic compounds with 3-hydrocarbyl-2-cyclopenten-1-ones followed by the dehydrative treatment of the intermediates with strong mineral acids

PA Targor G.m.b.H., Germany

SO Ger. Offen., 6 pp.

CODEN: GWXXBX

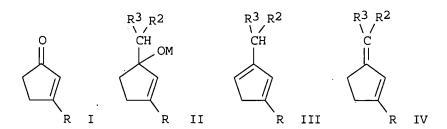
DT Patent

LA German

FAN.CNT 2

PATENT NO. KIND DATE APPLICATION NO. DATE

PI			DE 1999-19900732 19990112 WO 2000-EP15 20000104
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	·		DE 1999-19900732A 19990112 DE 1999-19935885A 19990730
PATE	NT FAMILY INFORM	ATION:	DB 1999 19993003A 19990730
FAN	2000:493491		
		KIND DATE	APPLICATION NO. DATE
PI			WO 2000-EP15 20000104
	-	CH, CY, DE, DK,	ES, FI, FR, GB, GR, IE, IT, LU, MC, NL,
			DE 1999-19900732A 19990112
	•		DE 1999-19935885A 19990730
			DE 1999-19900732 19990112
			DE 1999-19935885 19990730
			EP 2000-113883 20000630
	· · · · · ·		FR, GB, GR, IT, LI, LU, NL, SE, MC, PT,
	1E, S1,	LT, LV, FI, RO	DE 1000 10025005X 10000720
	TP 2001064210	X2 20010212	DE 1999-19935885A 19990730 JP 2000-215576 20000717
	OF 2001004210	A2 20010313	DE 1999-19935885A 19990730
			DE 1000 100000A 10000100



CASREACT 133:89249; MARPAT 133:89249

- AB In the title process, 3-hydrocarbyl-2-cyclopenten-1-ones (I; R = C1-20 hydrocarbyl, alkyl, aryl) (e.g., 3-methyl-2-cyclopenten-1-one) are reacted with organometallic compds. R2(R3)CHM [M = Li, MgCl, MgBr, MgI; R2, R3 = H, (un)branched alkyl, aryl] (e.g., n-BuLi), and the metal oxide intermediate (II) subjected to hydrolysis and dehydration by reaction with aq. solns. of strong mineral acids (e.g., aq. sulfuric acid) to give the desired "endo" product (III; e.g., 1-butyl-3-methylcyclopentadiene) and the undesired "exo" byproducts (IV).
- L16 ANSWER 9 OF 84 CAPLUS COPYRIGHT 2003 ACS on STN
- AN 1999:811200 CAPLUS
- DN 132:35338
- TI Process and catalysts for the symmetric disubstitution of carboxylic acid amides into substituted amines using Grignard reagents or organolithium compounds
- IN Buchholz, Herwig; Welz-Biermann, Urs; De Meijere, Armin
- PA Merck Patent GmbH, Germany
- SO PCT Int. Appl., 44 pp.

OS

GΙ

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CODEN: PIXXD2
DT
    Patent
LΑ
    German
FAN.CNT 8
                                        APPLICATION NO.
   PATENT NO.
                    KIND DATE
                                                        DATE
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                           19991223
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                                         DE 1998-19827161A 19980618
    DE 19827161
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    EP 1087933
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    EP 1088029
                     A1
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                                         EP 1999-932703 19990618
        R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, PT, FI
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                                         DE 1998-19827164A 19980618
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                                         DE 1998-19827166A 19980618
                                         DE 1998-19827167A 19980618
                                         DE 1998-19844194A 19980926
                                         WO 1999-EP4254 W 19990618
    JP 2002518365
                  Т2
                           20020625
                                         JP 2000-554689 19990618
                                         DE 1998-19827161A 19980618
                                         WO 1999-EP4254 W 19990618
    JP 2003524588
                  T2
                           20030819
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     Amides (e.g., diethylformamide) are sym. disubstituted on the geminal
AΒ
     carbonyl-C atom of the amide using organolithium compds. or Grignard
     reagents (e.g., phenylmagnesium bromide) to give substituted amines [e.g.,
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     optional organosilane (e.g., tert-butylsilyl trichloride) cocatalyst.
RE.CNT 6
              THERE ARE 6 CITED REFERENCES AVAILABLE FOR THIS RECORD
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L16 ANSWER 10 OF 84 CAPLUS COPYRIGHT 2003 ACS on STN
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     Method for catalytically disubstituting carboxylic acid amides with at
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IN
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    Carboxylic acid amides (e.g., diethylformamide) are disubstituted on the
    geminal carbonyl-C atom into an amine [e.g., N-(dicyclopentylmethyl)-N,N-
    diethylamine] in high yield and selectivity by the nucleophilic reaction
    of an amide using at least one Grignard reagent (e.g.,
    cyclopentylmagnesium bromide) in the presence of a metal alcoholate (e.g.,
     titanium tetraisopropoxide) used as a catalyst and in the presence of an
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             THERE ARE 15 CITED REFERENCES AVAILABLE FOR THIS RECORD
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     1999:811197 CAPLUS
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     Process and catalysts for the symmetrical disubstitution of carboxylic
     acid amides into substituted amines using Grignard reagents or
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     Buchholz, Herwig; Welz-Biermann, Urs
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     PCT Int. Appl., 34 pp.
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     carbonyl-C atom of the amide using organolithium compds. or Grignard
     reagents (e.g., phenylmagnesium bromide) to give substituted amines [e.g.,
     (diphenylmethyl)diethylamine], which reaction is conducted in the presence
     of a metal dioxide (e.g., titanium dioxide) catalyst and a metal
     alcoholate or organosilane (e.g., chlorotrimethylsilane) cocatalyst.
              THERE ARE 10 CITED REFERENCES AVAILABLE FOR THIS RECORD
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L16 ANSWER 12 OF 84 CAPLUS COPYRIGHT 2003 ACS on STN
AN
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    Method and catalysts for the disubstitution of carboxylic acid amides with
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IN
     Buchholz, Herwig; Welz-Biermann, Urs
PA
    Merck Patent G.m.b.H., Germany
SO
     PCT Int. Appl., 30 pp.
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    Amides (e.g., N-formylmorpholine) are disubstituted on the geminal
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    phenylmagnesium bromide and methylmagnesium bromide) to give substituted
     amines [e.g., 1-[(1-phenyl)ethyl]morpholine], which reaction is conducted
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              THERE ARE 9 CITED REFERENCES AVAILABLE FOR THIS RECORD
RE.CNT 9
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AN
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     Catalytic titanium(IV) dioxide-induced geminal asymmetric disubstitution
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     Buchholz, Herwig; Welz-Biermann, Urs
IN
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FAN	1999:811201 PATENT NO.	KIND DATE	APPLICATION NO. DATE
ΡI	WO 9965863 W: JP, KR,	US	WO 1999-EP4257 19990618
	RW: AT, BE,	CH, CY, DE, DK, ES	, FI, FR, GB, GR, IE, IT, LU, MC, NL,

PT, S	3		
DE 19827161 DE 19827163	A1	19991223	DE 1998-19827161A 19980618 DE 1998-19827163A 19980618 DE 1998-19827164A 19980618 DE 1998-19827165A 19980618 DE 1998-19827166A 19980618 DE 1998-19827167A 19980618 DE 1998-19844194A 19980926 DE 1998-19827161 19980618
DE 19827164	A1 A1	19991223 19991223	DE 1998-19827163 19980618 DE 1998-19827164 19980618
DE 19827165 DE 19827166	Al Al	19991223 19991223	DE 1998-19827165 19980618 DE 1998-19827166 19980618
DE 19844194	A1	19991223	DE 1998-19844194 19980926 DE 1998-19827167A119980618
WO 9965855 W: JP, K	A2	19991223	WO 1999-EP4255 19990618
•	E, CH, CY	, DE, DK, ES	, FI, FR, GB, GR, IE, IT, LU, MC, NL,
WO 9965318	A2	19991223	DE 1998-19827161A 19980618 DE 1998-19827163A 19980618 DE 1998-19827164A 19980618 DE 1998-19827165A 19980618 DE 1998-19827166A 19980618 DE 1998-19827167A 19980618 DE 1998-19844194A 19980926 WO 1999-EP4256 19990618
WO 9965318 W: JP, K	A3 R, US	20030417	•
·	E, CH, CY	, DE, DK, ES	, FI, FR, GB, GR, IE, IT, LU, MC, NL,
FD 1007020	7 .7	20010404	DE 1998-19827161A 19980618 DE 1998-19827163A 19980618 DE 1998-19827164A 19980618 DE 1998-19827165A 19980618 DE 1998-19827166A 19980618 DE 1998-19827167A 19980618 DE 1998-19844194A 19980926
EP 108.7929 R: AT, B	A1	20010404	EP 1999-929282 19990618
	s, CH, DE	, DK, ES, FR	, GB, GR, IT, LI, LU, NL, SE, PT, FI
	., Сн, DE	, DK, ES, FR	, GB, GR, IT, LI, LU, NL, SE, PT, FI DE 1998-19827161A 19980618 DE 1998-19827163A 19980618 DE 1998-19827164A 19980618 DE 1998-19827165A 19980618 DE 1998-19827166A 19980618 DE 1998-19827167A 19980618 DE 1998-19844194A 19980926 WO 1999-EP4257 W 19990618
JP 2002518366	T2	, DK, ES, FR	DE 1998-19827161A 19980618 DE 1998-19827163A 19980618 DE 1998-19827164A 19980618 DE 1998-19827165A 19980618 DE 1998-19827166A 19980618 DE 1998-19827167A 19980618 DE 1998-19844194A 19980926

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                                           DE 1998-19827164A 19980618
                                           DE 1998-19827165A 19980618
                                           DE 1998-19827166A 19980618
                                           DE 1998-19827167A 19980618
                                           DE 1998-19844194A 19980926
                                           WO 1999-EP4256 W 19990618
    US 2003009029
                      A1
                            20030109
                                           US 2002-162257
                                                           20020605
                                           DE 1998-19827164A 19980618
                                           US 2001-719810 A320010420
FAN
    1999:811202
     PATENT NO.
                      KIND
                            DATE
                                           APPLICATION NO.
                                                             DATE
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                            19991223
PΙ
    WO 9965864
                       A2
                                          WO 1999-EP4258
                                                             19990618
    WO 9965864
                       A3
                            20030522
        W: JP, KR, US
        RW: AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL,
             PT, SE
                                           DE 1998-19827161A 19980618
                                           DE 1998-19827163A 19980618
                                           DE 1998-19827164A 19980618
                                           DE 1998-19827165A 19980618
                                           DE 1998-19827166A 19980618
                                           DE 1998-19827167A 19980618
                                           DE 1998-19844194A 19980926
    DE 19827161
                       A1
                            19991223
                                           DE 1998-19827161 19980618
    DE 19827163
                            19991223
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                                           DE 1998-19827163 19980618
    DE 19827164
                       A1
                                           DE 1998-19827164 19980618
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    DE 19827165
                       A1
                            19991223
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    DE 19827166
                       A1
                            19991223
                                           DE 1998-19827166 19980618
    DE 19844194
                       A1
                            19991223
                                           DE 1998-19844194 19980926
                                           DE 1998-19827167A119980618
    WO 9965855
                                                             19990618
                       A2
                            19991223
                                           WO 1999-EP4255
            JP, KR, US
        RW: AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL,
             PT, SE
                                           DE 1998-19827161A 19980618
                                           DE 1998-19827163A 19980618
                                           DE 1998-19827164A 19980618
                                           DE 1998-19827165A 19980618
                                           DE 1998-19827166A 19980618
                                           DE 1998-19827167A 19980618
                                           DE 1998-19844194A 19980926
    WO 9965318
                            19991223
                                           WO 1999-EP4256
                       A2
                                                           19990618
    WO 9965318
                       A3
                            20030417
        W: JP, KR, US
        RW: AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL,
             PT, SE
                                           DE 1998-19827161A 19980618
                                           DE 1998-19827163A 19980618
                                           DE 1998-19827164A 19980618
                                           DE 1998-19827165A 19980618
                                           DE 1998-19827166A 19980618
                                           DE 1998-19827167A 19980618
                                           DE 1998-19844194A 19980926
    JP 2003524588
                            20030819
                                           JP 2000-554208
                                                            19990618
                                           DE 1998-19827161A 19980618
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DE 1998-19827164A 19980618
DE 1998-19827165A 19980618
DE 1998-19827166A 19980618
DE 1998-19827167A 19980618
DE 1998-19844194A 19980926
WO 1999-EP4256 W 19990618
US 2002-162257 20020605
DE 1998-19827164A 19980618
US 2001-719810 A320010420
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US 2003009029 A1 20030109

OS CASREACT 132:35334; MARPAT 132:35334

AB Amides (e.g., N-formylpiperidine) are disubstituted on the geminal carbonyl-C atom of the amide using two different Grignard reagents (e.g., phenylmagnesium bromide and ethylmagnesium bromide) to give substituted amines [e.g., 1-[(1-phenyl)propyl]piperidine], which reaction is conducted in the presence of a titanium dioxide catalyst and a metal alcoholate or organosilane (e.g., chlorotrimethylsilane) cocatalyst.

RE.CNT 6 THERE ARE 6 CITED REFERENCES AVAILABLE FOR THIS RECORD
ALL CITATIONS AVAILABLE IN THE RE FORMAT

L16 ANSWER 14 OF 84 CAPLUS COPYRIGHT 2003 ACS on STN

AN 1999:796156 CAPLUS

DN 132:151849

- TI Grignard reagent formation from aryl halides. There is no aryl radical intermediate along the dominant reaction channel
- AU Garst, J. F.; Ronald Boone, J.; Webb, L.; Easton Lawrence, K.; Baxter, J. T.; Ungvary, F.
- CS Department of Chemistry, The University of Georgia, Athens, GA, USA
- SO Inorganica Chimica Acta (1999), 296(1), 52-66 CODEN: ICHAA3; ISSN: 0020-1693
- PB Elsevier Science S.A.
- DT Journal
- LA English
- AB For Grignard reagent formation from Mg and an aliph. halide RX in an ether solvent, a route through R.bul. is the major pathway. Part of the evidence is that byproducts of side reactions of R.bul. are formed in substantial yields. Similar reactions of Ph and o-(3-butenyl)phenyl halides give very low (sometimes trace) yields of byproducts derived from side reactions of R.bul., despite the fact that aryl R.bul. are much more reactive than alkyl in both solvent attack and cyclization [o-(3-butenyl)phenyl case]. Grignard reactions of aryl halides appear to proceed largely through a pathway along which R.bul. is not an intermediate. This is probably a dianion pathway, i.e., one along which RX2- is an intermediate or transition state.
- RE.CNT 16 THERE ARE 16 CITED REFERENCES AVAILABLE FOR THIS RECORD ALL CITATIONS AVAILABLE IN THE RE FORMAT.
- L16 ANSWER 15 OF 84 CAPLUS COPYRIGHT 2003 ACS on STN
- AN 1999:425600 CAPLUS
- DN 131:44958
- TI Process for the manufacture of bis(phosphine oxide) and bis(phosphonate) compounds
- IN Foricher, Joseph; Schmid, Rudolf
- PA F. Hoffmann-La Roche A.-G., Switz.
- SO Eur. Pat. Appl., 17 pp. CODEN: EPXXDW
- DT Patent
- LA English

FAN.	CNT 1		
	PATENT NO.	KIND DATE	APPLICATION NO. DATE
PI		A1 19990630 B1 20020911	EP 1998-123996 19981217
		CH, DE, DK, ES, FR, LT, LV, FI, RO	GB, GR, IT, LI, LU, NL, SE, MC, PT,
			EP 1997-122720 A 19971223
	US 6162929	A 20001219	US 1998-212646 19981215
			EP 1997-122720 A 19971223
	AT 223923	E 20020915	AT 1998-123996 19981217
			EP 1997-122720 A 19971223
	ES 2182211	T3 20030301	ES 1998-123996 19981217
			EP 1997-122720 A 19971223
	JP 11246576	A2 19990914	JP 1998-364044 19981222
		,	EP 1997-122720 A 19971223
	CN 1224019	A 19990728	CN 1998-125786 19981223
			EP 1998-123996 A 19981217
OS GI	CASREACT 131:44	958; MARPAT 131:4495	

A process for the manuf. of bisphosphine oxide compds. I and II (R1, R2 = AB H, C1-8 alkyl, (un) substituted Ph, C1-8 alkoxy, phenyloxy, benzyloxy, halo, di-C1-8 alkylamino; R1R2 = fused ring, etc.; R3, R5 = H, C1-8 alkyl, (un) substituted Ph, C1-8 alkoxy, (un) substituted phenyloxy, benzyloxy, halo, di-C1-8 alkylamino; R4 = C1-8 alkoxy, (un) substituted phenyloxy, C1-8 alkyl, C3-7 cycloalkyl, (un) substituted Ph, naphthyl, heteroaryl, etc.; X = O, S) and bisphosphonates as intermediates for the prodn. of bisphosphine ligands, in which in a single step process (a) a phosphine oxide compd. is reacted in an org. solvent at -70.degree.-20.degree. with 0.5-3 equiv. of a lithium or magnesium amide compd., (b) 0.5-3 equiv. of oxidatively-acting metal salt or metal salt complex are added to the mixt. obtained in stage (a) in a temp. range of -70.degree.-20.degree., with a racemate of a bisphosphine oxide compd. being obtained; (c) a racemate cleavage is carried out if desired; and (d) the bisphosphonates obtained in stage (b) or (c) are converted into

9/24/2003>

bisphosphine oxides. Thus, **Grignard reaction** of 3-bromoanisole with P-chlorodiphenylphosphine in THF followed by H2O2 oxidn. gave 88.8% (3-methoxyphenyl)diphenylphosphine oxide. Coupling reaction of (3-methoxyphenyl)diphenylphosphine oxide in the presence of FeCl3 gave title compd. I (R1 = OMe, R2, R3 = H, R4 = Ph).

RE.CNT 3 THERE ARE 3 CITED REFERENCES AVAILABLE FOR THIS RECORD ALL CITATIONS AVAILABLE IN THE RE FORMAT

L16 ANSWER 16 OF 84 CAPLUS COPYRIGHT 2003 ACS on STN

AN 1999:407058 CAPLUS

DN 131:58656

TI Preparation of biphenyls having active substituents such as cyano group

IN Takahashi, Junya; Tsurushima, Masaaki

PA Mikuni Pharma Ind., Japan

SO Jpn. Kokai Tokkyo Koho, 9 pp.

CODEN: JKXXAF

DT Patent

LA Japanese

FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
ΡI	JP 11171799	A2	19990629	JP 1997-356313	19971208
				JP 1997-356313	19971208

Ι

OS CASREACT 131:58656; MARPAT 131:58656

GI

$$\mathbb{R} \xrightarrow{\text{CH}_2} \mathbb{R}$$

$$Y = CH_2$$

(A) n
 Z

II

Title compds. I [R = aryl, (aryl-contg.) hydrocarbyl; Z = active substituents; A = halo; k, m =0, 1; k + m = 1; n = 0-2] are prepd. by reaction of R(C6H4)kMgX (R, k = same as I; X = halo) with aryl compds. II (Z, A, n, m = same as I; Y = leaving group) in the presence of Cu salts. 4'-Bromomethylbiphenyl-4-carbonitrile was condensed with BuMgCl in the presence of Li2CuCl4 in THF at room temp. overnight to give 89.5% 4'-pentylbiphenyl-4-carbonitrile with 98.9% purity.

L16 ANSWER 17 OF 84 CAPLUS COPYRIGHT 2003 ACS on STN

AN 1999:311426 CAPLUS

DN 130:338554

Patel

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ΤI
     Preparation of substituted poly(arylenvinylenes) for use in
     electroluminescence
. IN
     Spreitzer, Hubert; Kreuder, Willi; Becker, Heinrich; Schenk, Hermann; Yu,
PA
     Hoechst A.-G., Germany
     Ger. Offen., 28 pp.
SO
     CODEN: GWXXBX
DT
     Patent
T.A
     German
FAN.CNT 1
     PATENT NO.
                      KIND DATE
                                           APPLICATION NO. DATE
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                            _____
                                            -----
                            19990506
                                           DE 1997-19748814 19971105
PΤ
     DE 19748814
                       A1
                       AA
                            19990520
                                            CA 1998-2308573 19981022
     CA 2308573
                                            DE 1997-19748814A 19971105
                                            WO 1998-EP6722 W 19981022
     WO 9924526
                            19990520
                                           WO 1998-EP6722
                       Α1
                                                             19981022
         W: CA, CN, JP, KR, MX, US
         RW: AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL,
                                            DE 1997-19748814A 19971105 ·
     EP 1029019
                       A1
                            20000823
                                            EP 1998-952737
                                                           19981022
         R: AT, CH, DE, FR, GB, IT, LI, NL
                                            DE 1997-19748814A 19971105
                                            WO 1998-EP6722 W 19981022
     JP 2001522926
                                            JP 2000-520525
                       T2
                             20011120
                                                             19981022
                                            DE 1997-19748814A 19971105
                                            WO 1998-EP6722 W 19981022
     US 2002064680
                       A1
                             20020530
                                            US 2000-530890
                                                             20000822
                                            DE 1997-19748814A 19971105
                                            WO 1998-EP6722 W 19981022
     US 2003088050
                       A1
                             20030508
                                            US 2002-185061
                                                             20020628
                                            DE 1997-19748814A 19971105
                                            WO 1998-EP6722 W 19981022
                                            US 2000-530890 B120000822
AB
     Poly(arylenevinylenes) bearing aryl substituents of specified structure,
     useful in electroluminescent lighting and displays, are prepd. Coupling
     di-Me bromoterephthalate with [3-[(3,7-dimethyloctyl)oxy]phenyl]boronic
     acid gave 98% di-Me 2-[3-[(3,7-dimethyloctyl)oxy]phenyl]terephthalate,
     redn. of which with LiAlH4 gave 82% corresponding bishydroxymethyl deriv.,
     treatment of which with SOC12 gave 70% 2,5-bis(chloromethyl)-3'-[(3,7-
     dimethyloctyl)oxy|biphenyl (I). Polymn. of 4.00 mmol each I and
     2,5-bis(chloromethyl)-3'-[(3,7-dimethyloctyl)oxy]-4-methoxybiphenyl in
     dioxane contg. tert-BuOK at 99.degree. gave 44% copolymer with wt.-av.
     mol. wt. 1,350,000. Use of the products in LED's is exemplified.
L16 ANSWER 18 OF 84 CAPLUS COPYRIGHT 2003 ACS on STN
     1997:448001 CAPLUS
AN
     127:67403
DN
ΤI
     Borate coinitiators for photopolymerization
IN
     Cunningham, Allan Francis; Kunz, Martin; Kura, Hisatoshi
     Ciba-Geigy A.-G., Switz.
PA
SO
     Ger. Offen., 41 pp.
     CODEN: GWXXBX
DT
     Patent
     German
T.A
FAN.CNT 2
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APPLICATION NO.

DATE

Patel 9/24/2003>

KIND DATE

PATENT NO.

·PI	DE 19648313	A1	19970528	DE 1996-19648313 19961121
			•	CH 1995-3342 A 19951124
	CH 691595	Α	20010831	CH 1996-2822 19961114
				CH 1995-3342 A 19951124
	AU 9671795	A1	19970529	AU 1996-71795 19961115
	AU 717137	B2	20000316	
				CH 1995-3342 A 19951124
•	FR 2741622	A1	19970530	FR 1996-14198 19961121
	FR 2741622	B1	19981204	
			,	CH 1995-3342 A 19951124
	CA 2191050	AA	19970525	CA 1996-2191050 19961122
	G. 2272000		233,0323	CH 1995-3342 A 19951124
	NL 1004597	A1	19970527	NL 1996-1004597 19961122
	NL 1004597	C2	19980107	112 1990 1001397 19901122
	112 1001337		13300101	CH 1995-3342 A 19951124
	GB 2307474	A1	19970528	GB 1996-24338 19961122
	GB 2307474	B2	19991215	05 1770 21330 17701122
	02 230,1,1	24	10001111	CH 1995-3342 A 19951124
	CN 1158854	Α	19970910	CN 1996-121743 19961122
	CN 1092199	В	20021009	CN 1990 121743 19901122
	CN 1032133		20021007	CH 1995-3342 A 19951124
	BE 1010761	A5	19990105	BE 1996-975 19961122
	22 1010,01	AJ	1000,0100	CH 1995-3342 A 19951124
	ES 2126499	A1	19990316	ES 1996-2464 19961122
	ES 2126499	B1	19991116	ES 1990-2404 19901122
	LD 2120499	DI	10001110	CH 1995-3342 A 19951124
	AT 9602040	Α	20000115	AT 1996-2040 19961122
•	AT 406775	В	20000115	A1 1990-2040 19901122
	111 400773	D	20000025	CH 1995-3342 A 19951124
	JP 09188685	A2	19970722	JP 1996-329208 19961125
	01 09100003	A2	177/0/22	CH 1995-3342 A 19951124
	BR 9605697	Α	19980818	BR 1996-5697 19961125
	BR 3003037	A	1000010	CH 1995-3342 A 19951124
	TW 494123	В	20020711	TW 1996-85114799 19961130
	111 494123	Ų	20020711	CH 1995-3342 A 19951124
DATE	NT FAMILY INFORMA	TTON.		CH 1995-5542
FAN	1999:582606	TION.		
I. VIIA	PATENT NO.	KIND	DATE	APPLICATION NO. DATE
	FAIBNI NO.	KIND	בות בות ב	APPLICATION NO. DATE
ΡI	US 5952152	A	19990914	US 1996-755380 19961121
	US 6210863	B1	20010403	US 1999-314391 19990519
			_0010100	CH 1995-3342 A 19951124
				US 1996-755380 A319961121
OC	MADDAT 127.67402			05 1770 733300 A317701121

OS MARPAT 127:67403

AB Monoborates of specified structure, bearing .gtoreq.2 aryl groups substituted in the ortho position, are very active coinitiators for photopolymn. The reaction of 60 mmol PhBr with 1.1 equiv. BuLi and 60 mmol fluorodimesitylborane in THF at room temp. gave 86% dimesitylphenylborane, reaction of which with MeLi in Et2O at 5.degree. followed by addn. of aq. Me4N+ Cl- gave 95% tetramethylammonium dimesitylmethylphenylborate (I). The use of I as a coinitiator in photocurable coatings is exemplified.

L16 ANSWER 19 OF 84 CAPLUS COPYRIGHT 2003 ACS on STN

AN 1996:739997 CAPLUS

DN 126:18658

TI Preparation of optically active biaryl compounds from 1-[2,6-

(bistrifluoromethanesulfonyloxy)phenyl]naphthalene and Grignard reagents

IN Hayashi, Tamio

PA Sumitomo Chemical Co, Japan

SO Jpn. Kokai Tokkyo Koho, 6 pp.

CODEN: JKXXAF

DT Patent

LA Japanese

FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
			-		
ΡI	JP 08245551	A2	19960924	JP 1995-51091	19950310
				JP 1995-51091	19950310

OS CASREACT 126:18658; MARPAT 126:18658

Ι

GI

AB Optically active biaryl compds. I (R = alkyl, alkenyl, aryl, aralkyl) are prepd. by treatment of I (R = CF3SO3) with RMgX (R = same as above; X = halo) in the presence of optically active metal complex catalysts prepd. from transition metal compds. and optically active R2R3NCHR1CH2PR42 [R1-R3 = lower alkyl, aryl, aralkyl; R4 = (cyclo)alkyl, alkoxy, (halo-substituted) Ph; NR2R3 may form ring]. I (R = CF3SO3) was treated with PhMgBr, LiBr, and (S)-PhCH2CH(NMe2)CH2PPh2-Pd complex at 0.degree. for 48 h to give 74% (S)-I (R = Ph) (84% e.e).

L16 ANSWER 20 OF 84 CAPLUS COPYRIGHT 2003 ACS on STN

AN 1996:264957 CAPLUS

DN 124:317470

TI Process for preparing silacyclohexane-based liquid crystal compounds

IN Kinsho, Takeshi; Shimizu, Takaaki; Ogihara, Tsutomu; Kaneko, Tatsushi; Nakashima, Mutsuo

PA Shin-Etsu Chemical Co., Ltd., Japan

SO Eur. Pat. Appl., 19 pp. CODEN: EPXXDW

DT Patent

LA English

FAN. CNT 1

L. M	N. CIVI I			
	PATENT NO.	KIND	DATE	APPLICATION NO. DATE
ΡI	EP 696591	A1	19960214	EP 1995-304845 19950711
	EP 696591	B1	20010314	
	R: DE, GB			
				JP 1994-182903 A 19940712
	JP 08081474	A2	19960326	JP 1995-198006 19950711
				JP 1994-182903 A 19940712
	US 5514824	Α	19960507	US 1995-501522 19950712

JP 1994-182903 A 19940712 EP 765880 **A**1 19970402 EP 1995-115429 19950929

R: DE, GB

JP 1994-182903 19940712

OS CASREACT 124:317470; MARPAT 124:317470

GI

AB A process for prepg. compds. I (R, Y, A = org. residues; i = 0-3) via reacting a ketone compd. II (Ar = org. residue) with an organometallic reagent and subsequently dehydrating, oxidizing, and Ar removal to give I is described. Thus, Grignard reaction of 4-(4-pentyl-4-phenyl-4-silacyclohexyl)-3-cyclohexenone with trans-(4-propylcyclohexyl)phenylmagnesium chloride in THF followed by p-toluenesulfonic acid-mediated dehydration and subsequent p-chloranil oxidative dehydrogenation gave 4-(4-pentyl-4-phenyl-4silacyclohexyl)-4'-(trans-4-propylcyclohexyl)biphenyl (III). Chlorination of III with BrCl in CCl4 followed by LiAlH4 redn. in THF gave title compd., trans, trans-4-(4-pentyl-4-silacyclohexyl)-4'-(4propylcyclohexyl)biphenyl. I are useful in liq. crystal displays.

ANSWER 21 OF 84 CAPLUS COPYRIGHT 2003 ACS on STN L16

AN 1996:11315 CAPLUS

DN 124:88136

ΤI Metallocene derivative catalyst components for polymerization of olefins, their manufacture, and manufacture of polyolefins using them

IN Murata, Masahide; Nakajima, Masashi; Kanazawa, Seizaburo; Ishihara, Takeshi; Ueki, Satoshi

PA Tonen Corp, Japan

Jpn. Kokai Tokkyo Koho, 12 pp. SO CODEN: JKXXAF

DT Patent

Japanese T.A

FAN.CNT 1

PATENT NO. KIND DATE APPLICATION NO. DATE -----_ _ _ _ PΙ JP 07268029 A2 19951017 JP 1994-81101 19940329 JP 1994-81101 19940329

MARPAT 124:88136 OS

AΒ The components are manufd. by prepg. metallocene derivs. contq. electron-donating groups, and by contacting them with Lewis-acid solid components. The polyolefins are manufd. by (co)polymg. olefins in the presence of the catalyst components and aluminoxanes. Thus, N, N-bis(trimethylsilyl)-3-bromopropylamine [prepd. from 3-bromopropylamine and N,N'-bis(trimethylsilyl)urea] and Cl2SiCp2 (Cp = cyclopentadienyl; prepd. from SiCl4 and CpLi) were Grignard reacted, treated with BuLi and ZrCl4, and desilylated to give a metallocene ligand, which was treated with a Mg-contg. solid carrier [prepd. from Mg, Bu20, I, BuCl, Et orthoformate, and SiCl4] to obtain a metallocene-supported catalyst. Ethylene was polymd. in the presence of 10 mg thus obtained catalyst and

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10085368.23

Page 54

Me aluminoxane to give a polymer with bulk d. 0.9 g/mL.

L16 ANSWER 22 OF 84 CAPLUS COPYRIGHT 2003 ACS on STN

AN 1994:162997 CAPLUS

DN 120:162997

TI Absolute stereochemistry of 1-(9-phenanthryl)-2-naphthoic acid as determined by CD and x-ray methods

AU Harada, Nobuyuki; Hattori, Tetsutaro; Suzuki, Takatsugu; Okamura, Atsuko; Ono, Hiroshi; Miyano, Sotaro; Uda, Hisashi

CS Inst. Chem. React. Sci., Tohoku Univ., Sendai, 980, Japan

SO Tetrahedron: Asymmetry (1993), 4(8), 1789-92 CODEN: TASYE3; ISSN: 0957-4166

DT Journal

LA English

GI

AB The abs. configurations of acid and alc. (R) - (-) - I (R = CO2H, CH2OH) both were assigned on the basis of CD data. This conclusion disagrees with previously assigned configurations based on the NMR of (S) -.alpha.-methylbenzyl 1-(9-phenthryl)-2-naphthoate [I; R = (S)-CO2CHMePh] (T. Suzuki, et al. 1990). To confirm the abs. configuration based on the CD method, an x-ray crystallog. structure anal. was carried out. The coupling reaction of (1R,3R,4S)-menthyl 1-[(1R,3R,4S)-menthyloxy]-2-naphthoate with 9-phenanthrylmagnesium bromide gave menthyl ester (S)-(+)-I (R = CO2R1, R1 = menthyl), which was hydrolyzed to afford acid (S)-(+)-I (R = CO2H) in 61% enantiomeric excess (ee). Acid chloride (S)-(+)-I (R = COCl) (61% ee) was reacted with the anion of (1S, 2R, 4R) - (-) - 2, 10-camphorsultam (R2-H), generated with sodium hydride. The diastereomeric mixt. of amides formed was sepd. by HPLC on silica gel. Amide (S)-(-)-I, (R = CO2R2) (II) was obtained as a major diastereoisomer from the first eluted fraction; redn. of II with LiAlH4 yielded alc. (S) - (+) - I (R = CH2OH). The second eluted fraction was evapd. to give amide (R) - (-) - I (R = CO2R2) (III), which was subjected to x-ray crystallog. anal.; the abs. configuration of III was assigned by the (1S, 2R, 4R) - (-) - 2, 10-camphorsultam part of the mol. From the chem. conversion and x-ray results the abs. configurations were detd.

L16 ANSWER 23 OF 84 CAPLUS COPYRIGHT 2003 ACS on STN

AN 1994:134337 CAPLUS

DN 120:134337

TI Synthesis of haloperidol ethanedithioketal HIV-1 protease inhibitors: magnesium chloride facilitated addition of Grignard reagents

Patel

AU Sui, Zhihua; De Voss, James J.; DeCamp, Dianne L.; Li, Jia; Craik, Charles S.; Ortiz de Montellano, Paul R.

CS Dep. Pharm. Chem., Univ. California, San Francisco, CA, 94143-0446, USA

SO Synthesis (1993), (8), 803-8 CODEN: SYNTBF; ISSN: 0039-7881

DT Journal

LA English

GΙ

$$S \longrightarrow (CH_2)_3N \longrightarrow X$$

AB Haloperidol ketals and ethanedithioketals, e.g. I (X = 0, OCH2CH2O), of interest as HIV-1 protease inhibitors were synthesized by addn. of organolithium and organomagnesium reagents to ketone precursors already contg. the ketal or thioketal functionality. Addn. of Grignard reagents to the thioketal contg. ketone was enhanced remarkably, and to the ketal contg. ketone moderately, by the addn. of magnesium chloride. The effect of magnesium chloride is attributed to its ability to competitively prevent chelation of the Grignard reagent and proton abstraction from the 4-oxopiperidine ring. The biol. activities of the ketals and thioketals indicate that the thioketal function conveys greater ability to inhibit the HIV-1 protease than the ketal function.

L16 ANSWER 24 OF 84 CAPLUS COPYRIGHT 2003 ACS on STN

AN 1994:107204 CAPLUS

DN 120:107204

Organomercury compounds. XXXI. Preparations and 199Hg NMR spectra of organomercury derivatives of 2-phenylpyridine, benzo[h]quinoline, 1-phenylpyrazole and 3,4,5-trimethyl-1-phenylpyrazole, and the x-ray crystal structure of bis[2-(pyridin-2'-yl)phenyl]mercury ,

AU Black, David St. C.; Deacon, Glen B.; Edwards, Gavin L.; Gatehouse, Bryan

CS Sch. Chem., Univ. New South Wales, Kensington, 2033, Australia

SO Australian Journal of Chemistry (1993), 46(9), 1323-36 CODEN: AJCHAS; ISSN: 0004-9425

DT Journal

LA English

OS CASREACT 120:107204

GI

AB 2-(Pyridin-2'-yl)phenylmercuric acetate has been prepd. by mercuration of 2-phenylpyridine. Symmetrization of the corresponding chloride by alk. sodium stannite gave bis[2-(pyridin-2'-yl)phenyl]mercury (I), which was also prepd. from 2-(2'-aminophenyl)pyridine by the diazo method and treatment of the initial product with copper powder and aq. ammonia. Mercuration of benzo[h] quinoline and 3,4,5-trimethyl-1-phenylpyrazole with mercuric acetate followed by treatment with lithium chloride yielded benzo[h]quinolin-10-ylmercuric chloride and 2-(3',4',5'-trimethylpyrazol-1'-yl)phenylmercuric chloride, Treatment of the former product with tribromide ions gave 10-bromobenzo[h]quinoline. The exchange Grignard reaction between 1-phenylpyrazole and ethylmagnesium bromide to give 2-(pyrazol-1'-yl)phenylmagnesium bromide has been monitored by reactions with benzonitrile and D2O to establish optimum conditions for reaction with mercuric bromide qiving bis[2-(pyrazol-1'-yl)phenyl]mercury. The 199Hq NMR chem. shifts of the majority of mercurials are shifted substantially downfield relative to the corresponding simple phenylmercurials consistent with weak intramol. coordination by the heterocyclic nitrogen donor atoms, but a small upfield shift is obsd. for bis[2-(pyrazol-1'-yl)phenyl]mercury. The x-ray crystal structure of I shows a centrosym. mol. with strong linear two coordination [Hg-C 2.098(8).ANG.; C-Hq-C 180.0.degree.] and significant but much weaker Hq-N interactions [Hg-N 2.798(7).ANG.; N-Hg-N 180.0.degree.] giving overall distorted square planar stereochem. The Ph rings are mutually coplanar, while the two pyridin-2'-yl rings are parallel and inclined at 10.8.degree. to the Ph groups.

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L16 ANSWER 25 OF 84 CAPLUS COPYRIGHT 2003 ACS on STN
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FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
ΡΙ	EP 501702 EP 501702 R: DE, FR,	A2 A3 GB, IT	19920902 19931208	EP 1992-301489	19920221
	R: DE, FR,	GB, 11		GB 1991-4050	19910227
	CA 2061770	AA	19920828	CA 1992-2061770	19920225
				GB 1991-4050	19910227
	JP 05086082	A2	19930406	JP 1992-76166	19920227
				GB 1991-4050	19910227
	US 5298663	Α	19940329	US 1993-35524	19930323
				GB 1991-4050	19910227
				US 1992-840353	19920224
	US 5414133 .	A	19950509	US 1994-179403	19940110
				GB 1991-4050	19910227
				US 1992-840353	1,9920224
				US 1993-35524	19930323

OS CASREACT 117:212725; MARPAT 117:212725

AN 1992:612725 CAPLUS

DN 117:212725

TI Process for the preparation of protected phosphine oxides from phosphinate esters and organomagnesium halides or organolithium compounds

IN Hall, Roger Graham; Riebli, Peter

PA Ciba-Geigy A.-G., Switz.

SO Eur. Pat. Appl., 11 pp. CODEN: EPXXDW

DT Patent

LA English

AB Protected phosphine oxides were prepd. by reaction of protected phosphinate esters with organomagnesium halides or organolithiums at -70 - +65.degree. Thus, EtOP(O)HCH2(OEt)2 was added to MeMgBr in THF at 0-5.degree. to give MeP(O)HCH2(OEt)2.

L16 ANSWER 26 OF 84 CAPLUS COPYRIGHT 2003 ACS on STN

AN 1992:550944 CAPLUS

DN 117:150944

TI Additions of 1-(.alpha.-aminoalkyl)benzotriazoles to enol ethers. New routes to 1,3-amino ethers

AU Katritzky, Alan R.; Rachwal, Stanislaw; Rachwal, Bogumila; Steel, Peter J.

CS Dep. Chem., Univ. Florida, Gainesville, FL, 32611-2046, USA

SO Journal of Organic Chemistry (1992), 57(18), 4932-9 CODEN: JOCEAH; ISSN: 0022-3263

DT Journal

LA English

OS CASREACT 117:150944

GI

AB 1-(.alpha.-Aminoalkyl)benzotriazoles add readily to enol ethers to give the corresponding 1-benzotriazolyl-3-aminoalkyl ethers, e.g. I (R1 = Ph, Me2CH) in high yields. Subsequent replacement of the benzotriazole moiety by an alkyl or aryl group (with a Grignard reagent) or by a hydrogen atom (with lithium aluminum hydride) affords 1,3-amino ethers in good yields. Anchimeric assistance by the amino groups in the substitutions of the benzotriazolyl moiety facilitates the reactions. Full stereochem. is assigned to the stereoisomeric products on the basis of NMR techniques and x-ray diffraction.

L16 ANSWER 27 OF 84 CAPLUS COPYRIGHT 2003 ACS on STN

AN 1992:531446 CAPLUS

DN 117:131446

TI Intramolecular aldol condensation applied to D-glucose-derived .delta.-ketoaldehydes: access to enantiomerically pure six-membered carbocycles

AU Tadano, Kinichi; Kanazawa, Satoshi; Takao, Kenichi; Ogawa, Seiichiro

CS Dep. Appl. Chem., Keio Univ., Yokohama, 223, Japan

SO Tetrahedron (1992), 48(21), 4283-300

CODEN: TETRAB; ISSN: 0040-4020

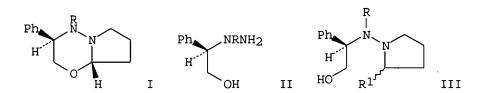
DT Journal

LA English

OS CASREACT 117:131446

GI

- * STRUCTURE DIAGRAM TOO LARGE FOR DISPLAY AVAILABLE VIA OFFLINE PRINT *
- AB An enantiomerically pure .delta.-ketoaldehyde I, efficiently prepd. from D-glucose, was subjected to an intramol. aldol condensation. The expected aldol reaction took place smoothly in the presence of DBU to give the aldol II stereoselectively. Further functionalization of II provided tri-C-substituted cyclohexanediols III and IV, via the functionalized cyclohexenone V.
- L16 ANSWER 28 OF 84 CAPLUS COPYRIGHT 2003 ACS on STN
- AN 1991:492231 CAPLUS
- DN 115:92231
- TI Organometallic reactions characteristic of chiral heterocyclic compounds: synthesis and stereoselective **Grignard reaction** of chiral 4-oxa-7,7a-diazaperhydroindans
- AU Takahashi, Hiroshi; Senda, Takashi; Higashiyama, Kimio
- CS Fac. Pharm. Sci., Hoshi Univ., Tokyo, 142, Japan
- SO Chemical & Pharmaceutical Bulletin (1991), 39(4), 836-42 CODEN: CPBTAL; ISSN: 0009-2363
- DT Journal
- LA English
- OS CASREACT 115:92231
- GI



- AB New heterocyclic compds., 6-phenyl-4-oxa-7,7a-diazaperhydroindans I (R = Me, CHMe2), were synthesized by condensation of chiral (2-hydroxyethyl)hydrazines II, prepd. from (R)-phenylglycinol, with .gamma.-chlorobutyraldehyde. The stereoselective Grignard reaction of I afforded chiral 2-substituted 1-[N-(2-hydroxy-1-phenylethyl)amino]pyrrolidines (III; R1 = Me, Et, Ph, CH2Ph). The mol. structure of I (R = Me) was detd. by x-ray crystallog. anal.
- L16 ANSWER 29 OF 84 CAPLUS COPYRIGHT 2003 ACS on STN
- AN 1991:255715 CAPLUS
- DN 114:255715
- TI Natural products syntheses using anodic oxidation of phenols as a key step
- AU Yamamura, Shosuke; Shizuri, Yoshikazu; Shigemori, Hideyuki; Okuno, Yoshishige; Ohkubo, Mitsuru
- CS Fac. Sci. Technol., Keio Univ., Yokohama, 223, Japan
- SO Tetrahedron (1991), 47(4-5), 635-44
 - CODEN: TETRAB; ISSN: 0040-4020
- DT Journal
- LA English
- AB Some bioactive natural products such as terpenoids, neolignans and others have been synthesized by means of an electrochem. method, wherein anodic

oxidn. of phenols is carried out as a key step.

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L16 ANSWER 30 OF 84 CAPLUS COPYRIGHT 2003 ACS on STN
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- AN 1991:247788 CAPLUS
- DN 114:247788
- TI Peptide derivatives preparation as retroviral protease inhibitors
- IN Kempf, Dale J.; Plattner, Jacob J.; Norbeck, Daniel W.; Boyd, Steven A.; Baker, William R.; Erickson, John W.; Fung, Anthony K. L.; Crowley, Steven R.
- PA Abbott Laboratories, USA
- SO PCT Int. Appl., 222 pp.

CODEN: PIXXD2

DT Patent

LA English

FAN.CNT 1

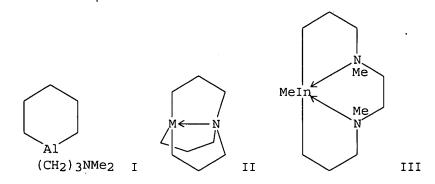
	PATENT NO.			KIND	DATE	APPLICATION NO. DATE	
ΡI	WO	8910' W:	-		A1 JP, KR,		WO 1989-US2055 19890512
		RW:	AT,	BE,	CH, DE	FR, GB,	, IT, LU, NL, SE
							US 1988-194678 19880513
	ΕP	3425	41		A2	19891123	EP 1989-108590 19890512
	ΕP	3425	41		A3	19911106	5
		R:	ES,	GR			
							US 1988-194678 19880513
	ΑU	8935	660		A1	19891129	AU 1989-35660 19890512
							US 1988-194678 19880513
							WO 1989-US2055 19890512
	EΡ	4159	81		A1	19910313	EP 1989-905856 19890512
		R:	AT,	BE,	CH, DE,	, FR, GB,	, IT, LI, LU, NL, SE
							US 1988-194678 19880513
							WO 1989-US2055 19890512
	JР	0350	4247		T2	19910919	JP 1989-506033 19890512
							US 1988-194678 19880513
							WO 1989-US2055 19890512

OS MARPAT 114:247788

AB Peptide derivs. are prepd. as retroviral protease inhibitors. Synthetic processess involved carbodiimide coupling, or coupling in combination with deprotection, and reaction with mixed anhydrides. Thus, N-methyl-1-cyclohexenecarboxamide was treated with BuLi in THF, treated with ClTi(OPr-iso)3, and then Boc-phenylalaninal to give N-methyl-6-[2-(tert-butoxycarbonyl)amino-1-hydroxy-3-phenyl]propyl-1-cyclohexenecarboxamide. This was then deprotected with HCl in dioxane to give N-methyl-6-(2-amino-1-hydroxy-3-phenylpropyl)-1-cyclohexenecarboxamide-HCl (I). I was coupled with Boc-Leu-Asn in the presence of 180-BuO2CCl to give the amide.

- L16 ANSWER 31 OF 84 CAPLUS COPYRIGHT 2003 ACS on STN
- AN 1991:247347 CAPLUS
- DN 114:247347
- TI Intramolecular, metallacyclic organoaluminum, -gallium and -indium addition compounds. Crystal structure of 1-galla-5-azabicyclo[3.3.3]undecane
- AU Schumann, Herbert; Hartmann, Uwe; Wassermann, Wilfried; Just, Oliver; Dietrich, Andreas; Pohl, Ludwig; Hostalek, Martin; Lokai, Matthias
- CS Inst. Anorg. Anal. Chem., Tech. Univ. Berlin, Berlin, W-1000/12, Germany
- SO Chemische Berichte (1991), 124(5), 1113-19 CODEN: CHBEAM; ISSN: 0009-2940

DT Journal LA English GI



- AB The prepn. of title compds., such as I, II (M = Al, Ga), and III, was described. Thus, the reaction of Me2N(CH2)3AlCl2 with BrMg(CH2)5MgBr gave I in 49% yield. III was prepd. in 47% yield from MeInCl2 and ClMg(CH2)3NMeCH2CH2NMe(CH2)3MgCl. An x-ray anal. of II (M = Ga) was described. I and 2 other products were successfully tested as vapor-phase epitaxy precursors.
- L16 ANSWER 32 OF 84 CAPLUS COPYRIGHT 2003 ACS on STN
- AN 1991:143424 CAPLUS
- DN 114:143424
- TI Processes for the preparation of naphthalenylmethyl-3H-1,2,3,5-oxathiadiazole 2-oxides as antihyperglycemic agents
- IN Ellingboe, John W.; Bagli, Jehan F.; Alessi, Thomas R.
- PA American Home Products Corp., USA
- SO U.S., 15 pp.

CODEN: USXXAM

- DT Patent
- LA English

FAN.CNT 1

	U1.1 1				
	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
ΡI	US 4966975	Α	19901030	US 1989-341615	19890421
				US 1989-341615	19890421
~~	MADDAM 114 14040			•	

OS MARPAT 114:143424

GI

AB The title compds. (I; R = H, alkyl, alkoxy, halo), useful for treatment of diabetes mellitus (no data), were prepd. from RC6H4CH2CO2H by 1) reaction with SOCl2, 2) cyclocondensation of the resulting acid chloride

Patel

with C2H4 in the presence of TiCl4 or AlCl3, 3) treatment of the resulting 2-tetralone with MeMgBr, MeCeCl2, or MeTiCl3, 4) aromatization of the resulting naphthol with Ph3COH in CF3CO2H or with Ph3CBF4, 5) halogenation of the obtained 2-naphthalene, 6) treatment of the obtained 2-halomethylnaphthalene with Li-, Na-, or KCN, 7) condensation of the cyanomethyl compd. with NH2OH, and 8) cyclocondensation of the imidamide with SOCl2. Title compd. II was prepd. by the above method starting from 2-BrC6H4CH2CO2H; addnl. methods start from 2-naphthoic acid.

L16 ANSWER 33 OF 84 CAPLUS COPYRIGHT 2003 ACS on STN

AN 1991:114595 CAPLUS

DN 114:114595

TI Novel heteroarotinoids: synthesis and biological activity

AU Spruce, Lyle W.; Gale, Jonathan B.; Berlin, K. Darrell; Verma, A. K.; Breitman, Theodore R.; Ji, Xinhua; Van der Helm, Dick

CS Dep. Chem., Oklahoma State Univ., Stillwater, OK, 74078, USA

SO Journal of Medicinal Chemistry (1991), 34(1), 430-9

CODEN: JMCMAR; ISSN: 0022-2623

DT Journal

LA English

OS CASREACT 114:114595

GI

I, R=Me, $R^1=C_6H_4CO_2Me-4$

II, R=Me, $R^1=C_6H_4CO_2H-4$

IV, R=H, R 1 =E, \dot{E} -CH=CHCMe=CO $_2$ H

V, R=Me, $R^1=E$, Z-CH=CHCMe=CO₂H

VII, $R=R^1=Me$, X=S

VIII, R=Me, R^1 =H, X=S

IX, R=H, $R^1=Me$, X=S

 $X, R=R^1=H, X=S$

XI, R=H, $R^1=Me$, X=O

 CO_2R^1 XII, $R=R^1=H$, X=O

AB Thirteen heteroarotinoids were synthesized. The key step in each prepn. was the condensation of the appropriate chroman-, thiochroman-, or benzothienyl-substituted phosphorus ylide, obtained from the independent synthesis of the corresponding phosphonium salts, with selected polyene-substituted aldehyde esters. Screening of the compds. was with one of two assays. One assay measured the ability of a retinoid to inhibit the phorbol ester induced increase of mouse epidermal ornithine decarboxylase (ODC) activity. The other assay measured retinoid-induced differentiation of the human myeloid leukemia cell line HL-60. In the ODC assay, all thirteen compds. were screened. The most active

Patel

heteroarotinoids were ester I and the acid II. Both of these retinoids had ID50 values (dose required for half-maximal inhibition of phorbol ester induced ODC activity) of about 0.3 nmol. In comparison, the ID50 value for trans-retinoic acid III was 0.12 nmol while the ID50 values for acids IV and V were about 3.5 nmol. Heteroarotinoids VI and VII-XII had ID50 values of 35 nmol or greater. With a thiochroman unit, the most active acids in decreasing order of activity in the ODC assay were II > V > VI. Thus, simple replacement of the terminal propenyl system [C(16,17,18)] in IV with a cyclopropyl group produced acid VI with markedly reduced activity. With a benzoic acid group as part of the structure attached to the thiochroman unit, the ODC activity was enhanced as shown in I and II. The combination of the 2,2,4,4tetramethylthiochroman group and the benzoic acid (or ester) terminal group seemed to enhance the biol. action which resembles that found with (E) -4-[2-(5,6,7,8-tetrahydro-5,5,8,8-tetramethyl-2-naphthalenyl)-1propenyl]benzoic acid, a well-known model system. Replacing the protons with fluorine in the C(12) Me group in the side chain and altering the orientation of the aryl groups around the double bond from anti to syn lowered ODC activity in both the thiochroman- and chroman-contq. systems. Esters VII and IX and acid VIII were essentially inactive while acid X exhibited a high ID50 in the ODC assay. In the chroman family, both ester XI and acid XII had unfavorable ID50 values. Since acid VIII differs only slightly from acid X [the latter is devoid of the geminal di-Me group at C(2)] and acid X differs only slightly from acid XII, possibly the nature of the heteroatom and the stereochem. at the .alpha. position may play important roles in regulating activity, but more examples are required to establish a trend. Changing the ring size from a fused six-six system to a five-six system led to ester Me (E)-4-[2-(2,3-dihydro-3,3dimethylbenzo[b]thien-5-yl)-1-propenyl]benzoate (XIII) and acid (E)-4-[2-(2,3-dihydro-3,3-dimethylbenzo[b]thieny-5-yl)-1-propenyl]benzoic acid (XIV), resp. In sep. expts. from those with of I-XII and known compds both XIII and XIV exhibited similar inhibition of ODC activity to that of III at the 34 nmol level. The ID50 values of XIV and XIV were, however, 10 and 200 times greater than that of III resp. In view of the toxicity of III, ester XIII may hold promise in chemotherapy. Of eight heteroarotinoids examd. in the HL-60 assay system, only acid IV displayed modest activity. This acid had an ED50 value (dose required for half-maximal effect) of 500 nM. In comparison, the ED50 for III was 50 nM. All of the other heteroarotinoids had ED50 values which were greater than 1000 nM.

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L16 ANSWER 34 OF 84 CAPLUS COPYRIGHT 2003 ACS on STN
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- AN 1991:82316 CAPLUS
- DN 114:82316
- TI Total synthesis of avermectin Bla: synthesis of the carbohydrate bis-oleandrose fragment and coupling to the avermectin Bla aglycon
- AU Ford, Mark J.; Knight, Julian G.; Ley, Steven V.; Vile, Sadie
- CS Dep. Chem., Imp. Coll. Sci., Technol. Med., London, SW7 2AY, UK
- SO Symlett (1990), (6), 331-2 CODEN: SYNLES; ISSN: 0936-5214
- DT Journal
- LA English
- OS CASREACT 114:82316

GI

- AB Oleandrose (I, R = R1 = H)(II) was prepd. in 8 steps from (S)-Me3CSiMe2OCHMeCHO via cyclic sulfites III (4 isomers) and the key intermediate .pi.-allyl tricarbonyl iron complexes IV and V. Acetylation of II gave an equimolar mixt. of diacetate I (R = R1 = Ac) and monoacetate I (R = Ac, R1 = H) (VI). The diacetate was selectively deacetylated with LiBEt3H to give monoacetate I (R = H, R1 = Ac), which was coupled via its imidazolylcarbonyl deriv., with VI to give the bis-oleandrose fragment VII. VII was selectively deacetylated with LiBEt3H, treated with thiocarbonyldiimidazole and then coupled with avermectin Bla aglycon monoacetate in the presence of AgClO4 and K2CO3 to give avermectin Bla diacetate. The latter compd. was deacetylated with excess LiBEt3H to give avermectin Bla.
- L16 ANSWER 35 OF 84 CAPLUS COPYRIGHT 2003 ACS on STN
- AN 1990:630521 CAPLUS
- DN 113:230521
- TI Mechanistic aspects of the ligand-assisted nucleophilic addition reaction
- AU Swiss, Kevin A.; Liotta, Dennis C.; Maryanoff, Cynthia A.
- CS Dep. Chem., Emory Univ., Atlanta, GA, 30322, USA
- SO Journal of the American Chemical Society (1990), 112(25), 9393-4 CODEN: JACSAT; ISSN: 0002-7863
- DT Journal
- LA English
- AB A systematic study of Grignard addns. to quinol alkoxides elucidates the origins of the high facial selectivity obsd. in these 1,4 addn. processes. The reactive intermediate is a ternary complex composed of a quinol alkoxide, a dialkylmagnesium, and a Lewis acid.
- L16 ANSWER 36 OF 84 CAPLUS COPYRIGHT 2003 ACS on STN
- AN 1990:532280 CAPLUS
- DN 113:132280
- TI Regio- and stereoselective synthesis of allyltrimethylsilanes via Krief-Reich elimination in .beta.-seleno-.gamma.-silyl alcohols
- AU Sarkar, Tarun K.; Ghosh, Sunil K.; Satapathi, Tushar K.
- CS Dep. Chem., Indian Inst. Technol., Kharagpur, 721 302, India
- SO Tetrahedron (1990), 46(6), 1885-98 CODEN: TETRAB; ISSN: 0040-4020

DT Journal

LA English

OS CASREACT 113:132280

AB The synthesis of (E)-allyltrimethylsilanes by regio- and stereocontrolled pathways is described based on the preference for Krief-Reich elimination over silicon-controlled rearrangement in .beta.-seleno- .gamma.-silyl alcs., readily available from .alpha.-selenoaldehydes. Usefulness of this protocol for the introduction of the allylsilane function .alpha. to the carbonyl group in cycloalkanones as well as for the prepn. of unsym. substituted allylsilanes is also reported.

L16 ANSWER 37 OF 84 CAPLUS COPYRIGHT 2003 ACS on STN

AN 1990:440676 CAPLUS

DN 113:40676

TI 1-[(2-fluorophenyl)(4-fluorophenyl)phenylmethyl]-1H-imidazole and related medical fungicides

IN Bartroli, Javier; Anguita, Manuel

PA Uriach, J., y Cia. S. A., Spain

SO Eur. Pat. Appl., 19 pp. CODEN: EPXXDW

DT Patent

LA English

FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
ΡI	EP 352352	A1	19900131	EP 1988-112239	19880728
	EP 352352 ·	B1 .	19911016		•
	R: AT, BE,	CH, DE	, FR, GB, GR,	IT, LI, LU, NL, SE	
	AT 68486	E	19911115	AT 1988-112239 ·	19880728
				EP 1988-112239	19880728
	JP 02048569	A2	19.900219	JP 1988-268344	19881026
	JP 06025145	B4	19940406		
				EP 1988-112239	19880728
	CA 1327589	A1	19940308	CA 1988-582916	19881114
	·			EP 1988-112239	19880728
	US 5149707	A	19920922	US 1990-628937	19901214
				EP 1988-112239	19880728
				US 1988-257095	19881013

OS CASREACT 113:40676; MARPAT 113:40676

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9/24/2003>

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AB The title compd. (I) (UR-4506) (and related compds.) were prepd. by reaction of [(2-fluorophenyl)(4-fluorophenyl)phenyl]chloromethane (II) (or the correspong trityl chlorides) with imidazole or Li imidazolide in a polar, inert solvent (MeCN) at 0.degree.-reflux for 5 min-3 h. Thus, PhMgBr reacted with 2,4'-difluorobenzophenone to give 97% of the tritylcarbinol, which was converted to II (83%) with SOCl2. II was stirred with Li imidazolide in MeCN at room temp. for 2 h to give 96% I. I had an MIC of 1.0 .mu.g/mL against Candida albicans ATCC 10231. I formulations are given.

L16 ANSWER 38 OF 84 CAPLUS COPYRIGHT 2003 ACS on STN

AN 1990:439715 CAPLUS

DN 113:39715

TI Photochemistry of 4-acylisoxazoles

AU Sauers, Ronald R.; Hadel, L. M.; Scimone, A. A.; Stevenson, T. A.

CS New Brunswick Dep. Chem., Rutgers, State Univ., New Brunswick, NJ, 08903, USA

SO Journal of Organic Chemistry (1990), 55(13), 4011-19 CODEN: JOCEAH: ISSN: 0022-3263

DT Journal

LA English

OS CASREACT 113:39715

GI

The photochem. of 4-acylisoxazoles (I, II, III, IV, V) was investigated in a effort to clarify literature contradictions and anomalies and to provide a more detailed picture of the nature and no. of intermediates involved in photoreactions of these systems. In contrast to a previous report on the photorearrangement of IV, both oxazoles expected from a 2H-azirine intermediate have been obsd. Wavelength studies of II and the derived 2H-azirine revealed evidence for the involvement of at least two distinct product-forming intermediates. The results of quantum yield and laser flash photolysis measurements for ketones I, IV and V have been interpreted in terms of rapid ring openings of ketone triplet states to form diradical-like intermediates coupled with efficient reclosures (70-99%).

L16 ANSWER 39 OF 84 CAPLUS COPYRIGHT 2003 ACS on STN

AN 1990:98163 CAPLUS

DN 112:98163

TI Reactions of 2-phenylethyl and 3-phenylpropyl carbinols with fluorosulfuric acid

- AU Bright, Steven T.; Coxon, James M.; Steel, Peter J.
- CS Dep. Chem., Univ. Canterbury, Christchurch, N. Z.
- SO Journal of Organic Chemistry (1990), 55(4), 1338-44 CODEN: JOCEAH; ISSN: 0022-3263
- DT Journal
- LA English
- OS CASREACT 112:98163
- AB A series of 2-phenylethyl and 3-phenylpropyl carbinols have been reacted with HSO3F at -78 degree., the solns. quenched, and the products isolated to give good yields of cyclization products. The 2-phenylethyl carbinols generally undergo rearrangement prior to cyclization, whereas the 3-phenylpropyl carbinols undergo direct cyclization of the initially formed carbocation, to give tetralins. The mechanisms and synthetic applications of these reactions are discussed.
- L16 ANSWER 40 OF 84 CAPLUS COPYRIGHT 2003 ACS on STN
- AN 1990:97722 CAPLUS
- DN 112:97722
- TI Preparation of ketones by reaction of Grignard reagents with acid halides using **lithium** tetrahalometallate catalysts
- IN Sproesser, Linhard; Sperling, Karin; Trautmann, Walter; Smuda, Hubert
- PA BASF A.-G., Fed. Rep. Ger.
- SO Ger. Offen., 8 pp. CODEN: GWXXBX
- DT Patent
- LA German
- FAN CNT 1

LHIA.	~TA T	Τ.										
	PA	ΓENT	NO.		KII	ND.	DATE			API	PLICATION NO.	DATE
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PI	DE	3744	619		A:	1 .	1989	0713		DE	1987-3744619	19871231
	ΕP	3257	84		A2	2	1989	0802		EP	1988-121614	19881223
	ΕP	3257	84		A.	3	1990	0704				
		R:	ΒĒ,	CH,	DE,	FR,	GB,	IT,	LI,	NL		
										DE	1987-3744619	19871231

- OS MARPAT 112:97722
- AB Ketones, including intermediates for agrochems., are prepd. by reaction of carboxylic acid halides with Grignard reagents in an inert solvent with added LiMnX4, LiFeX4, or Li2CuX4 (X = F, Cl, Br, or I). Thus, a Mg-free, 1.2-M soln. of Me3CMgCl in THF (150 mL) under N at 10.degree. was treated with 10.5 mL of a 0.5-M soln. of Li2MnCl4 in THF, followed by 0.075 mol Ph0(CH2)3COCl in THF. Hydrolysis with aq. NH4Cl and extn. gave 14 g crude (75% purity) Ph0(CH2)3COCMe3, purifiable by distn.
- L16 ANSWER 41 OF 84 CAPLUS COPYRIGHT 2003 ACS on STN.
- AN 1989:614170 CAPLUS
- DN 111:214170
- TI Derivatives of tamoxifen. Dependence of antiestrogenicity on the 4-substituent
- AU McCague, Raymond; Leclercq, Guy; Legros, Nicole; Goodman, Joyce; Blackburn, G. Michael; Jarman, Michael; Foster, Allan B.
- CS Drug Dev. Sect., Inst. Cancer Res., Sutton/Surrey, SM2 5PX, UK
- SO Journal of Medicinal Chemistry (1989), 32(12), 2527-33 CODEN: JMCMAR; ISSN: 0022-2623
- DT Journal
- LA English
- OS CASREACT 111:214170

GI

AB A range of tamoxifen (I; R = H) derivs. substituted in the 4-position of the 1-Ph ring are described. The key steps in the synthesis of I (R = iodo, Br, MeS) were reactions of 1,2-diarylbutanones with the (4-halophenyl)lithium or [4-(methylthio)phenyl] magnesium bromide. Oxidized precursors of 4-(methylthio)tamoxifen were used to prep. the methylsulfinyl and methylsulfonyl derivs. Further derivs. I (R = formyl, hydroxymethyl, oxiranyl, mercapto) were prepd. from 4-bromotamoxifen via the 4-lithio deriv. Several of the derivs. I (R = Br, iodo, SMe, SOMe, SO2Me, oxiranyl, CHO, CH2OH) displayed a higher affinity for estrogen receptors (ER) of calf uterine cytosol than did tamoxifen, but there was no relationship between affinity to ER and the ability to inhibit the growth of the MCF-7 breast cancer cell line in vitro.

L16 ANSWER 42 OF 84 CAPLUS COPYRIGHT 2003 ACS on STN

AN 1989:553242 CAPLUS

DN 111:153242

TI Addition and redox processes in the reaction of Grignard reagents with 1,4-dinitrobenzene. Factors affecting product distribution

AU Bartoli, Giuseppe; Dalpozzo, Renato; Grossi, Loris

CS Dip. Sci. Chim., Camerino, 62032, Italy

Ι

SO Journal of the Chemical Society, Perkin Transactions 2: Physical Organic Chemistry (1972-1999) (1989), (6), 573-8
CODEN: JCPKBH; ISSN: 0300-9580

DT Journal

LA English

OS CASREACT 111:153242

ΑB 1,4-Dinitrobenzene (I) reacts smoothly and irreversibly with alkylmagnesium or -lithium reagents to give at first the nitroarene radical anion (redox product) and 6-alkyl-2-nitro-5-acinitrocyclohexa-1,2-diene (II) (addn. product). Intermediate II undergoes an immediate addn. to the nitro function by a second mole of Grignard reaction or MeLi to give trans-4,5-dialkyl-3,6di-aci-nitrocyclohexene, which can be converted into the corresponding trans-5,6-dialkyl-1,4-dinitrocyclohexa-1,3-diene by oxidn. with NaOCl or DDQ. The addn. process is favored by lower temps. and weekly polar and highly viscous solvents, while steric hindrance in the magnesium reagent enhances radical anion formation. These findings are interpreted in terms of a single electron transfer mechanism in which all factors delaying a geminate recombination of the radical pair favor the redox process to the detriment to addn. The almost abs. stereoselectivity of the double alkylation process is attributed to steric control on the direction of attack of alkylmagnesium to the ene-nitro function of II exerted by the axial alkyl group. A detailed ESR study of the radical

anion of I is also reported.

L16 ANSWER 43 OF 84 CAPLUS COPYRIGHT 2003 ACS on STN

AN 1989:534015 CAPLUS

DN 111:134015

TI Preparation of (E)-N,N-dimethyl-3-[6,11-dihydrodibenzo[b,e]thiepin-11-ylidene]propylamine as an antidepressant

IN Polivka, Zdenek; Protiva, Miroslav

PA Czech.

SO Czech., 4 pp.

CODEN: CZXXA9

DT Patent

LA Czech

FAN.CNT 1

				CS 1986-6447	19860905
ΡI	CS 254742	В1	19880115	CS 1986-6447	19860905
	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE

OS CASREACT 111:134015

GΙ

The title thiepin (I; R = CH2NMe2), the trans isomer of prothiaden, was prepd. as antidepressant (no data) by reducing I (R = CONMe2) (II) with LiAlH4 in boiling Et2O. A soln. of 26 g II (multistep prepn. given) in 150 mL Et2O was added to a suspension of 5 g LiAlH4 in 200 mL Et2O and refluxed 4 h. The product was isolated, redissolved in EtOH and treated with a soln. of HCl in Et2O to give 24 g I.HCl. M.p. of I.HCl was 227-229.degree. (from EtOH/Et2O); I m. 52-54.degree.

L16 ANSWER 44 OF 84 CAPLUS COPYRIGHT 2003 ACS on STN

AN 1989:154889 CAPLUS

DN 110:154889

TI Preparation of norstatine- and norcyclostatine-containing peptides as renin inhibitors

IN Hoover, Dennis Jay; Wester, Ronald Thure; Rosati, Robert Louis

PA Pfizer Inc., USA

SO Eur. Pat. Appl., 86 pp. CODEN: EPXXDW

DT Patent

LA English

FAN. CNT 1

L'ATA.	CIAI	_														
	PA:	CENT	NO.		KI	ND	DATE			A	PLIC	CATI	ON NO	0.	DATE	
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ΡI	ΕP	2669	50		A.	2	1988	0511		E	9 198	37-3	0946	1	19871	027
	ΕP	2669	50		A.	3	1990	0411								
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		R:	ΑT,	ΒE,	CH,	DE,	ES,	FR,	GB,	GR,	IT,	LI,	LU,	NL,	SE	

T.V. 150056	_	10040115	US 1986-925449 A 19861031 US 1987-68982 A 19870701
IN 172976	A	19940115	IN 1987-DE905 19871015 US 1986-925449 19861031
CN 87101499	A	19880511	CN 1987-101499 19871023
CN 1027271	В	19950104	US 1986-925449 A 19861031
	_	1000001	US 1987-68982 A 19870701
US 4814342	Α	19890321	US 1987-112976 19871023
			US 1986-925449 A219861031 US 1987-68982 A219870701
AT 99324	E	19940115	AT 1987-309461 19871027
A1 55521	-	17710113	US 1986-925449 A 19861031
			US 1987-68982 A 19870701
•			EP 1987-309461 A 19871027
ES 2061512	Т3	19941216	ES 1987-309461 19871027
			US 1986-925449 A 19861031
			US 1987-68982 A 19870701
CA 1310793	A1	19921124	CA 1987-550413 19871028
			US 1986-925449 A 19861031
DV 0705604	7	10000501	US 1987-68982 A 19870701 DK 1987-5684 19871030
DK 8705684	Α	19880501	DK 1987-5684 19871030 US 1986-925449 A 19861031
			US 1987-68982 A 19870701
FI 8704787	Α	19880501	FI 1987-4787 19871030
FI 90346	В	19931015	11 150, 170, 250, 1030
FI 90346	С	19940125	
			US 1986-925449 A 19861031
NO 8704530	Α	19880502	NO 1987-4530 19871030
NO 173017	В	19930705	
NO 173017	С	19931013	HC 1006 025440 N 10061021
			US 1986-925449 A 19861031 US 1987-68982 A 19870701
AU 8780541	A1	19880505	AU 1987-80541 19871030
AU 585180	· B2	19890608	
			US 1986-925449 A 19861031
			US 1987-68982 A 19870701
HU 45270 HU 207869	A2 B	19880628	HU 1987-4901 19871030
NO 207669	ь	19930628	US 1986-925449 A 19861031
			US·1987-68982 A 19870701
JP 63183551	A2	19880728	JP 1987-275583 19871030
			US 1986-925449 A 19861031
			US 1987-68982 A 19870701
DD 262583	A 5	19881207	DD 1987-308473 19871030
73 0700150	_	10000000	US 1986-925449 A 19861031
ZA 8708158	Α	19890628	ZA 1987-8158 19871030 US 1986-925449 A 19861031
SU 1706391	A3	19920115	SU 1987-4203604 19871030
50 1,00331	110	13320113	US 1986-925449 A 19861031
US 4935405	Α	19900619	· US 1988-277614 19881129
			US 1986-925449 B219861031
			US 1987-68982 B219870701
110 5024254	-	10010500	US 1987-112976 A319871023
US 5034376	Α	19910723	US 1990-497041 19900321
			. US 1986-925449 B219861031 US 1987-68982 B219870701
			US 1987-112976 A319871023

		•		HS	1988-277614	Δ319881129
IN	175148	Α	19950506	ΙN	1990-DE781	19900803
				IN	1987-DE905	A119871015
JP	07173134	A2	19950711	JP	1994-221930	19940916
JP	07108901	B4	19951122			
				US	1986-925449	A 19861031
	•			US	1987-68982	A 19870701

OS CASREACT 110:154889; MARPAT 110:154889

GI

AΒ The title peptides [I, II; Z = R1-Ym-Ap; R1 = C1-6 alkyl, C1-4 alkoxy, (un) substituted amino, morpholino, piperidyl, piperazino, (substituted)piperidino, thiomorpholino, pyridyl, etc; Y = CO, P(O)OMe, SO2; A = NMe, NH, O; m, p = 0, 1; M = Ph, PhCH2, naphthyl, thienyl, MeOC6H4, ClC6H4, HoC6H4, C6-7 cycloalkyl; X = Me, H; R2 = C1-5 alkyl, substituted C1-2 alkyl, PhCH2, guanidino-C1-3 alkyl, 4-aminobutyl, imidazol-4-ylmethyl, etc.; X = cyclohexyl, Me2CH, Ph; W = CHOH, CO, CHN3, CHNH2, CMeOH, etc.; Z1 = CH2OH, R-X1-T; R = CO; X1 = O, NH, NMe, CH2, bond; T = C1-5 alkyl, C1-4 hydroxyalkyl, C1-4 alkylcarbamoyl, H, trifluoroethyl, Ph, PhCH2, morpholino, etc.; L = CH, N; R5 = imidazol-4-ylmethyl, C2-5 alkyl; R6 = C1-4 alkoxy, C1-4 alkylamino; provided that when m = O, P = O; when A = O, Y = CO; when T = C1-4alkylcaRbamoyl, X1 = NH, NMe, CH2; when T = C2-5 alkylamino, C1-2 alkoxyamino, morpholino or 4-C1-2 alkylpiperazino, X1 = CH2, bond], useful as antihypertensives (no data), were prepd. Treatment of (S)-3-(tert-butoxycarbonylamino)-4-cyclohexyl-(R)-2-hydroxybutyric acid with Me2CHCH2O2CCl in THF contg. Et3N and amidation of the resulting mixed anhydride with MeNH2 gave 42% N-methyl-3-(tert-butoxycarbonylamino)-4cyclohexyl-(R)-2-hydroxybutyramide (BOC-nor-C-Sta-NHMe). Deprotection of the latter with 4N HCl in dioxane, followed by peptide coupling with BOC-Phe-His(imBOC)-OH (BOC = CO2CMe3) in CH2Cl2 in the presence of Et3N, hydroxybenzotriazole, and DCC, gave BOC-Phe-His(imBOC)-nor-C-Sta-NHMe, which was treated with AcOH-H2O(80:20) to give BOC-Phe-His-nor-C-Sta-NHMe.

L16 ANSWER 45 OF 84 CAPLUS COPYRIGHT 2003 ACS on STN

AN 1989:24095 CAPLUS

DN 110:24095

- General synthesis of ketones from carboxylic esters and carboxamides by TT use of mixed organolithium-magnesium reagents: syntheses of artemisia ketone
- ΑU
- Fehr, Charles; Galindo, Jose; Perret, Roland Res. Lab., Firmenich S. A., Geneva, CH-1211/8, Switz. CS
- Helvetica Chimica Acta (1987), 70(7), 1745-52 SO CODEN: HCACAV; ISSN: 0018-019X
- Journal DT
- LΑ English
- OS CASREACT 110:24095
- The novel reagents formed by combination of Grignard reagents with AB LiN(CHMe2)2 convert non-enolizable or slowly enolizable carboxylic esters or carboxamides into ketones which are protected from further reaction by their in situ conversion into enolates. These enolates were trapped with electrophiles such as Me3SiCl and BrCH2CH:CH2. The scope of the Grignard mono-addn. is illustrated by 2 direct syntheses of artemisia ketone.
- ANSWER 46 OF 84 CAPLUS COPYRIGHT 2003 ACS on STN L16
- AN 1989:24018 CAPLUS
- DN 110:24018
- Synthesis of heterocyclic compounds containing germanium and nitrogen as TI hetero-atoms. I
- AU · Shitara, Kazuhiro; Sato, Yoshiro; Nakagawa, Reiko
- CS Fac. Pharm. Sci., Nagoya City Univ., Nagoya, 467, Japan
- Journal of Organometallic Chemistry (1988), 339(3), 259-65 SO CODEN: JORCAI; ISSN: 0022-328X
- DT Journal
- LΑ English
- OS CASREACT 110:24018
- GI

- AB Several new 5- and 6-membered heterocyclic compds. contg. both Ge and N were synthesized starting from Me2Ge(CH2Cl)Cl (I). Thus, Grignard reaction of 2-MeC6H4MgBr with I gave 2-MeC6H4GeMe2CH2Cl which on bromination with NBS gave 2-BrCH2C6H4GeMe2CH2Cl (II). Cyclocondensation of II with RNH2 gave 73-95% tetrahydrobenzoazagermines III (R = Me, Ph, Me2NCH2CH2CH2). Grignard reaction of I with H2C: CHMgBr gave Me2Ge (CH2Cl) (CH: CH2) which on treatment with R1NH2 gave 50-93% Me2Ge(CH2NHR1)(CH:CH2) (IV; R1 = Ph, PhCH2). Reductive cyclization of IV gave azagermolidines V. Azaoxadigermines VI (R2 = Et, PhCH2) were prepd. by the hydrolysis of I with NaHCO3 followed by cyclocondensation with R2NH2. Some reactions of VI were also described.
- L16 ANSWER 47 OF 84 CAPLUS COPYRIGHT 2003 ACS on STN
- 1988:493295 CAPLUS AN
- DN 109:93295
- TΙ Structural characterization in solutión of intermediates in

rhodium-catalyzed hydroformylation and their interconversion pathways

- AU Brown, John M.; Kent, Alexander G.
- CS Dyson Perrins Lab., Oxford, OX1 3QY, UK
- SO Journal of the Chemical Society, Perkin Transactions 2: Physical Organic Chemistry (1972-1999) (1987), (11), 1597-607 CODEN: JCPKBH; ISSN: 0300-9580
- DT Journal
- LA English
- OS CASREACT 109:93295

GI

- AB The reaction of HRh(CO)(PPh3)3 (I) with CO has been studied by 1H, 13C, and 31P NMR. The main species present under ambient conditions is HRh(CO)2(PPh3)2 (II) which exists as two rapidly equilibrating trigonal bipyramidal isomers. Complexes I and II are in rapid equil. via CO and PPh3 dissocn. steps and the square-planar complexes HRh(CO)(PPh3)2 (III) and HRh(CO)2PPh3 (IV) are likely transient intermediates. The chem. of these PPh3 complexes is compared with that of closely related 5-phenyl-5H-dibenzophosphole and 1,3-bis(diphenylphosphino)propane analogs. Complex I catalyzes the isomerization of (Z)-[1,2-2H2]styrene, effectively suppressed by CO or PPh3. HRh(CO)2P2 complexes trap methylenecyclopropane. In the presence of styrene and CO, I is converted into a branched acyl deriv., e.g. V, which readily equilibrates with its linear isomer VI; the stereochem. of these acyl derivs. is detd. by low-temp. NMR; at higher temps. rapid inter- and intramol. exchange processes occur. The relevance of these observations to Rh-catalyzed hydroformylation is discussed and it is proposed that the regiochem. of reaction is largely controlled by competitive olefin trapping involving complexes III and IV.
- L16 ANSWER 48 OF 84 CAPLUS COPYRIGHT 2003 ACS on STN
- AN 1988:6285 CAPLUS
- DN 108:6285
- TI Preparation of new 5.alpha.-hydroxy-.DELTA.9(10)-19-norsteroids and their conversion to .DELTA.4-19-norsteroids useful as antiquococrticoids
- IN Philibert, Daniel; Teutsch, Jean Georges; Costerousse, Germain; Deraedt,
 Roger
- PA Roussel-UCLAF , Fr.
- SO Fr. Demande, 61 pp. CODEN: FRXXBL
- DT Patent
- LA French
- FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
ΡI	FR 2586021	A1	19870213	FR 1985-12216	19850809
	FR 2586021	B1	19881014		
				FR 1985-12216	19850809

GΙ

$$R^{1}$$
 R^{2}
 R^{3}
 R^{5}
 R^{5}
 R^{5}
 R^{5}

5.alpha.-Hydroxy-19-norsteroids I [R1 = alkyl, alkenyl, furyl, cycloalkyl, AΒ naphthyl, di-Ph, (un) substituted thienyl or Ph; R2 = Me, Et; R3 = H, OH, HOCH2CO, carboxyalkoxy, acyloxyalkyl, (un)substituted alkyl, alkenyl, alkynyl, (un)ketalized Ac, and R4 = H, OH, CH2CN, (un)substituted alkyl, alkenyl, alkynyl; or R3 = cyano and R4 = ether-protected OH; R5 = H, .alpha.- or .beta.-Me; K = keto group blocked as a ketal, thicketal, oxime, or methyloxime; various further provisos are given] are prepd. and converted to the 19-norsteroids II [X = O, NOH, alkoxyimino; AB = O, bond; similar R-groups and provisos], which are antiglucocorticoids. A soln. of 3,3-ethylenebis(oxy)-5.alpha.,10.alpha.-epoxy-17.alpha.-(prop-1-ynyl)estr-9(11)-en-17.beta.-ol in THF was treated with a soln. of Cu reagent (from CuCl and 4-MeSC6H4MgBr) in THF, and the mixt. was stirred for 2 h at -20. degree. to give I [R1 = 4-MeSC6H4, R2 = Me, R3 = OH, R4 = C.tplbond.CMe, R5 = H, K = OCH2CH2O]. Deprotection and dehydration of the latter by refluxing in 95% EtOH with the acidic sulfonate resin Redex CF gave the corresponding II (X = 0, AB = bond, others as given) (III). Tablets of 120 mg each contained 50 mg III and the remainder of talc, starch, and Mg stearate. III had a 24-h relative binding affinity 227% that of dexamethasone for isolated rat thymus glucocorticoid receptors.

L16 ANSWER 49 OF 84 CAPLUS COPYRIGHT 2003 ACS on STN

AN 1987:636318 CAPLUS

DN 107:236318

TI Total synthesis of (.+-.)-citreoviridin

AU Williams, David R.; White, F. H.

CS Dep. Chem., Indiana Univ., Bloomington, IN, 47405, USA

SO Journal of Organic Chemistry (1987), 52(23), 5067-79

CODEN: JOCEAH; ISSN: 0022-3263

DT Journal

LA English

OS CASREACT 107:236318

GI

AB Studies detailing the stereochem. course of iodine-induced cyclization of .gamma.,.delta.-olefinic benzyl ethers I (R = R1 = H, OH; R2 = R3 = H, Me; R4 = MeOCH2CH2OCH2) affording tetrahydrofurans II (R5 = CHIMe) were described. In all cases, the stereochem. of a secondary allylic hydroxyl influenced the course of ring closure to position the new .alpha.-iodoethyl substituent in an anti (trans) disposition relative tot he neighboring alc. Unambiguous stereochem. assignments were available from x-ray crystallog. studies of II (R = H, R1 = OH). The key intermediates (.+-.)-citreoviral (II; R = OH, R1 = R4 = H, R5 = trans-CH:CMeCHO) (III) was prepd. by Wittig methodol. in 6 steps from II (R = OH, R1 = H, R4 = MeOCH2CH2OCH2, R5 = CHIMe). III was converted to (.+-.)-citreoviridin (IV) by extension of the olefinic chain and introduction of the .alpha.-pyrone unit.

L16 ANSWER 50 OF 84 CAPLUS COPYRIGHT 2003 ACS on STN

AN 1987:631464 CAPLUS

DN 107:231464

TI Preparation of unsaturated amide pesticides

IN Blade, Robert John; Parkin, Donald; Crombie, Leslie; Horsham, Mark Andrew

PA Wellcome Foundation Ltd., UK

SO Eur. Pat. Appl., 29 pp.

CODEN: EPXXDW

DT Patent

LA English

FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	EP 228222 EP 228222 EP 228222	A3	19870708 19890426 19900523	EP 1986-309742	19861215
	R: AT, BE,			GR, IT, LI, LU, NL GB 1985-31073 GB 1986-19983	, SE 19851217 19860815
	AU 8666567	A1	19870618	AU 1986-66567	19861215
	AU 604660	B2	19910103		
				GB 1985-31073 GB 1986-19983	19851217 19860815
	FI 8605097	A	19870618	FI 1986-5097 GB 1985-31073	19861215 19851217

Patel

			GB 1986-19983	19860815
DK 8606031	Α	19870618	DK 1986-6031	19861215
			GB 1985-31073	19851217
			GB 1986-19983	19860815
HU 42261	A2	19870728	HU 1986-5214	19861215
			GB 1985-31073	19851217
			GB 1986-19983	19860815
JP 62187441	A2	19870815	JP 1986-298592	19861215
			GB 1985-31073	19851217
			GB 1986-19983	19860815
BR 8606203	Α	19870929	BR 1986-6203	19861215
			GB 1985-31073	19851217
			GB 1986-19983	19860815
ZA 8609437	Α	19880727	ZA 1986-9437	19861215
			GB 1985-31073	19851217
AT 53009	Ē	19900615	AT 1986-309742	19861215
			GB 1985-31073	19851217
			GB 1986-19983	19860815
			EP 1986-309742	19861215

AB The unsatd. amides QX(CH2)m(CY:CY1)nCONRR1 [X = CH2, O; m = 0-10; n = 1, 2; Y, Y1 = H, alkyl, haloalkyl; R, R1 = H, (un)substituted alkyl, aminoalkyl, alkenyl, alkynyl, or cycloalkyl; Q = alkoxy, cycloalkyl, (un) substituted alkyl, etc.] are prepd. as insecticides, acaricides and fungicides. Tri-Et 4-phosphonocrotonate in dry THF was added at -70.degree. to Li diisopropylamide in dry THF, followed by 7,7-difluorohept-6-enal (prepn. given) at -60.degree. to give Et (2E,4E)-11,11-difluoroundeca-2,4,10-trienoate, which was refluxed in KOH-contg. EtOH for 2 h, to give the corresponding acid. This was treated with Et3N and with Ph N-phenylphosphoramidochloridate in dry CH2Cl2, followed by iso-BuNH2 and Et3N in CH2Cl2, to give (2E,4E)-N-isobutyl-11,11difluoroundeca-2,4,10-trienamide (I). I had a topical LD50 value of 0.5 .mu.g/fly against houseflies (Musca domestica) when synergized with piperonyl butoxide. A spray was made of I 0.1, butylated hydroxyanisole 0.1, xylene 10.0, and kerosene 89.8 (no units given).

L16 ANSWER 51 OF 84 CAPLUS COPYRIGHT 2003 ACS on STN

AN 1987:407398 CAPLUS

DN 107:7398

TI Synthesis of some monoterpenols via cyclopropylcarbinyl rearrangement

AU Moiseenkov, A. M.; Ceskis, B.

CS N. D. Zelinskii Inst. Org. Chem., Moscow, USSR

SO Collection of Czechoslovak Chemical Communications (1986), 51(6), 1316-22 CODEN: CCCCAK; ISSN: 0366-547X

DT Journal

LA English

OS CASREACT 107:7398

GΙ

AB Treating bromodimethylcyclopropane with Li in hexane followed by DMF gave

- 2,2-dimethylcyclopropanecarboxaldehyde which underwent <code>Grignard reactions</code> with CH2:CHC(:CH2)MgCl and CH2:CMeCHMgCl to give stereoisomeric alcs. I and II. The latter were cleaved by HClO4 to give Me2C(OH)CH2CH:CHC(:CH2)CH:CH2 and Me2C(OH)CH2CH:CHCH2CMe:CH2, resp. Similarly obtained were MeC(OH)CHMeCH:CHCHMe2 and Me2C(OH)CHMeCH:CHCMe:CH2.
- L16 ANSWER 52 OF 84 CAPLUS COPYRIGHT 2003 ACS on STN
- AN 1987:102459 CAPLUS
- DN 106:102459
- TI Electron-deficient pentamethylcyclopentadienyl-1,3-butadiene complexes of titanium, zirconium, and hafnium
- AU Blenkers, Joop; Hessen, Bart; Van Bolhuis, Fre; Wagner, Anton J.; Teuben, Jan H.
- CS Dep. Chem., Univ. Groningen, Groningen, 9747 AG, Neth.
- SO Organometallics (1987), 6(3), 459-69 CODEN: ORGND7; ISSN: 0276-7333
- DT Journal
- LA English
- OS CASREACT 106:102459
- Cp*M(diene)Cl [I; Cp* = C5Me5; M = Ti, Zr, Hf; diene = CH2:CRCR1:CH2; R = AB R1 = H, Me (L); R = Me, R1 = H] were prepd. by either redn. of Cp*MCl3 (M = Zr, Hf) in the presence of free diene, reaction of Cp*TiCl3 with the enediylmagnesium reagent [Mg(CH2CMe:CMeCH2)].cntdot.2THF, or exchange of an .eta.3-1-methallyl ligand between Cp*M(butadiene)(1-methallyl) and Cp*MCl3. These 14-electron complexes form 16-electron adducts with a variety of Lewis bases, in all of which the diene ligand assumes a nonfluxional s-cis conformation. NMR spectroscopy indicates diene-metal bonding of .sigma.2,.pi.-metallacyclopentene rather than .eta.4-diene character. EHMO calcns. on 14- and 16-electron model systems predict less pronounced metallacyclopentene character on complexation of the Lewis base. Both types of compds. were characterized by x-ray crystallog. diene C-C distances in Cp*Hf(C6H10)Cl (II) indicate a large participation of the asym. .eta.3, .sigma.-resonance structure in the bonding of the diene fragment to the metal. The metallacyclopentene character of the diene ligand in II.cntdot.py is apparent from the Hf-C(diene) and diene C-C distances. The Cl atom in I is easily substituted to form alkyl, aryl, allyl, and borohydride derivs. I show agostic C-H-M interactions in NMR and IR spectra.
- L16 ANSWER 53 OF 84 CAPLUS COPYRIGHT 2003 ACS on STN
- AN 1987:101701 CAPLUS
- DN 106:101701
- TI Perfluoroalkylations of carbanions with (perfluoroalkyl)phenyliodonium triflates (FITS reagents)
- AU Umemoto, Teruo; Gotoh, Yoshihiko
- CS Sagami Chem. Res. Cent., Kanagawa, 229, Japan
- SO Bulletin of the Chemical Society of Japan (1986), 59(2), 439-45 CODEN: BCSJA8; ISSN: 0009-2673
- DT Journal
- · LA English
- OS CASREACT 106:101701
- AB F(CF2)nIPhO3SCF3 (I, n = 2, 3, 4, 6, 8) reacted with alkylmagnesium halides, e.g., Me(CH2)7MgCl, to give up to 82% perfluoroalkylated alkanes, e.g., Me(CH2)7(CF2)7CF3. However, similar treatment of I with secondary or tertiary alkylmagnesium halides, aryl or vinyl magnesium halides, and alkyllithium or -copper compds. gave low yields of perfluoroalkylated products. RC.tplbond.CLi (R = Ph, hexyl) reacted with

I (n = 2, 3, 6, 8) to give 44-70% RC.tplbond.C(CF2)nF. I also reacted smoothly with enolate anions of active methylene compds., e.g., 2-methyl-1,3-cyuclopentanedione, MeCOCHMeCO2Et, and Et 2-oxocyclopentanecarboxylate in polar solvents to afford O- and C-perfluoroalkylation product in moderate yields. The ratio of O to C products was temp. dependent. Metalated MeCH(CO2Et)2 and Me2CHNO2 gave C-perfluoroalkylated products only.

L16 ANSWER 54 OF 84 CAPLUS COPYRIGHT 2003 ACS on STN

AN 1987:49637 CAPLUS

DN 106:49637

10085368.23

TI Synthesis of (E)-1-propenyl ketones from carboxylic esters and carboxamides by use of mixed organolithium-magnesium reagents.

Synthesis of .alpha.-damascone, .beta.-damascone, and .beta.-damascenone

AU Fehr, Charles; Galindo, Jose

CS Res. Lab., Firmenich SA, Geneva, CH-1211, Switz.

SO Helvetica Chimica Acta (1986), 69(1), 228-35 CODEN: HCACAV; ISSN: 0018-019X

DT Journal

LA English

OS CASREACT 106:49637

GI

E-Propenyl ketones, e.g., I, were prepd. in yields as high as .apprx.85% by Grignard reaction of esters and carboxamides (e.g., II, R = MeO, Et2N) with CH2:CHCH2MgCl in THF-hexane in the presence of R21NLi (R1 = Me2CH, Et), followed by isomerization with p-MeC6H4SO3H in PhMe. A mechanism involving ketone protection via its enolate is supported by the formation of silyl enol ether III in the reaction of II (R = Et2N) with CH2:CHCH2MgCl and (Me2CH)2NLi when quenched with Me3SiCl.

L16 ANSWER 55 OF 84 CAPLUS COPYRIGHT 2003 ACS on STN

AN 1984:490365 CAPLUS

DN 101:90365

TI Preparation of chiral sulfones by asymmetric addition of arenesulfonyl carbanions to acetone

AU Akiyama, Takahiko; Shimizu, Makoto; Mukaiyama, Teruaki

CS Fac. Sci., Univ. Tokyo, Tokyo, 113, Japan

SO Chemistry Letters (1984), (4), 611-14

CODEN: CMLTAG; ISSN: 0366-7022

DT Journal

LA English

OS CASREACT 101:90365

9/24/2003>

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- AB In the presence of a chiral diamine, the magnesium salt of allyl p-tolyl sulfone reacted with acetone at the .alpha.-carbon to give 4-MeC6H4SO2CH(CH:CH2)CMe2OH. Chiral 1-substituted 2-(aminomethyl)pyrrolidines were used.
- L16 ANSWER 56 OF 84 CAPLUS COPYRIGHT 2003 ACS on STN
- AN 1984:156178 CAPLUS
- DN 100:156178
- TI Preparation of allylic and homoallylic alcohols containing trifluoromethyl group
- AU Koh, Moon Gyu; Choi, Sam Kwon
- CS Dep. Chem., Korea Adv. Inst. Sci. Technol., Seoul, 131, S. Korea
- SO Bulletin of the Korean Chemical Society (1983), 4(5), 200-3 CODEN: BKCSDE; ISSN: 0253-2964
- DT Journal
- LA English
- AB Treatment of alkynols RC.tplbond.CCH(OH)CF3 (R = Bu, Ph) with Me2CHCH2MgCl in the presence of Cp2TiCl2 (Cp = cyclopentadienyl) in ether afforded allylic alcs. (Z)-RCH:CHCH(OH)CF3. Reaction of the (E)-type Grignard intermediates with MeI or iodine gave (Z)-MeCR:CHCH(OH)CF3 and (E)-ICR:CHCH(OH)CF3. Phenylation of CH2:CHZCH(OH)CF3 (Z = bond, CH2) with PhI in the presence Pd(OAc)2 gave (E)-PhCH:CHXCH(OH)CF3.
- L16 ANSWER 57 OF 84 CAPLUS COPYRIGHT 2003 ACS on STN
- AN 1981:586837 CAPLUS
- DN 95:186837
- TI Substituted benzyl esters of cyclopropane carboxylic acids compositions containing them and methods of combating insect pests therewith, and substituted benzyl alcohols
- IN Punja, Nazim
- PA Imperial Chemical Industries Ltd. , UK
- SO Eur. Pat. Appl., 63 pp. CODEN: EPXXDW
- DT Patent
- LA English
- FAN.CNT 3

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				GB 1979-44151	19791221
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FAN		32:562446						
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				GB	1979-44151	19791221
			•	GB	1980-37257	19801120
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				GB	1979-44151	19791221
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				GB	1980-40400	19801217
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GI

Cyclopropanecarboxylates I [R1, R2 = Me, halomethyl, halo; R3 = alkyl, alkenyl, PhCH2, H; X = O, S, SO2, NR4 (R4 = H, alkyl, carboxylic acyl); m = 0, 1; n = 1-4], useful as insecticides and acaricides, were prepd. by 4 methods. C-Methylating 1,2,3,4-F4C6H2 with BuLi and MeI at -60 to -45.degree. gave 2,3,4,5-F4C6HMe which was carboxylated with successive addns. of BuLi and CO2 (g) at -70.degree. to -40.degree. to give 3,4,5,6-F4C6(CO2H)Me-2,1. This was reduced (LiAlH4 in Et2O) to 3,4,5,6-F4C6(CH2OH)Me-2,1 which was esterified with 50:50 cis- to trans-cyclopropanecarbonyl chloride II to give 50:50 cis- to trans-I [Fn = 3,4,5,6-F4, (X)mR3 = 2-Me](III). At 50 ppm, III killed 90-100% Musca domestica and I [Fn = 2,3,5,6-F4, (X)mR3 = 4-allyl] killed 100% Tetranychus tetarius.

L16 ANSWER 58 OF 84 CAPLUS . COPYRIGHT 2003 ACS on STN

AN 1981:569342 CAPLUS

DN 95:169342

TI Synthesis and properties of bis(pentamethylcyclopentadienyl) actinide hydrocarbyls and hydrides. A new class of highly reactive f-element

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organometallic compounds

- ΑU Fagan, Paul J.; Manriquez, Juan M.; Maatta, Eric A.; Seyam, Afif M.; Marks, Tobin J.
- IL, USA CS
- Journal of the American Chemical Society (1981), 103(22), 6650-67 SO CODEN: JACSAT; ISSN: 0002-7863
- DT Journal
- English LΑ
- ΑÈ The synthesis, chem., and physicochem. properties of thorium and uranium bis(pentamethylcyclopentadienyl) chlorides, hydrocarbyls, chlorohydrocarbyls, and hydrides were reported. The reaction of the precursor compds. M[.eta.5-(CH3)5C5]2Cl2 with 2 equiv of lithium reagent RLi produces M[.eta.5-(CH3)5C5]2R2 (R = CH3, CH2Si(CH)3, CH2C(CH3)3, CH2C6H5, and C6H5, M = Th; R = CH3, CH2Si(CH3)3, CH2C6H5, C6H5, M = U) in high yield. With 1 equiv of lithium reagent, M[.eta.5-(CH3)5C5]2(R)C1 (R = CH2C(CH3)3, CH2Si(CH3)3, CH2C6H5, C6H5, M =Th; R = CH2C(CH3)3, CH2Si(CH3)3, CH2C6H5, C6H5 M = U) are formed in high yield. The M[.eta.5-(CH3)5C5]2(CH3)Cl compds. can be synthesized by redistribution between the corresponding di-Me and dichloro complexes. The new organoactinides were thoroughly characterized by elemental anal., 1H NMR and vibrational spectroscopy, and in many cases cryoscopic mol. wt. measurements. The hydrocarbyls and chlorohydrocarbyls generally exhibit high thermal stability. However, the di-Ph compds. react readily with C6D6 to yield, via a benzyne complex, the corresponding M(C6D5)2 compds. The thorium bis(neopentyl) complex reacts with benzene to produce the corresponding di-Ph complex. In probes of bond polarity, the di-Me complexes react rapidly with acetone, alcs., and iodine to produce resp. the corresponding tert-butoxides, alkoxides plus methane, and iodides plus Competition expts. at -78.degree. indicate that the thorium complexes are more reactive than those of uranium. M[.eta.5-(CH3)5C5]2C5]2R2 compds. undergo hydrogenolysis to yield organoacetinide hydrides, {M[.eta.5-(CH3)5C5]2(.mu.-H)H}2, and RH. While the thorium hydride exhibits high thermal stability, that of uranium readily (and reversibly) eliminates H2, forming a U(III) hydride. hydrides react vigorously with CH3Cl to produce methane and the corresponding chloro complexes, with acetone to produce isopropoxy complexes, and with alcs. to produce alkoxides and H2. The thorium chlorohydride, {Th[.eta.5-(CH3)5C5]2(.mu.-H)Cl}2, can be prepd. by redistribution of the dichloride and dihydride; an alkoxyhydride, Th[.eta.5-(CH3)5C5]2[OC(CH3)3]H, can be prepd. by hydrogenolysis of Th[.eta.5-(CH3)5C5][OC(CH3)3]CH3. In soln., the metal-bound hydrides of {Th[.eta.5-(CH3)5C5]2(.mu.-H)H}2 rapidly exchange with dissolved H2; this hydride also reacts with ethylene to yield the corresponding di-Et complex. The olefin addn. and hydrogenolysis reactions can be coupled to effect homogeneous, catalytic olefin hydrogenation. The differences between thorium and uranium chem. appear largely to reflect differences in accessible oxidn. states and in metal-ligand bond polarity.
- L16 ANSWER 59 OF 84 CAPLUS COPYRIGHT 2003 ACS on STN
- AN 1981:561132 CAPLUS
- DN 95:161132
- TIPreparation of highly reactive metal powders. New procedure for the preparation of highly reactive zinc and magnesium metal powders
- ΑU Rieke, Reuben D.; Li, Percy Tzu-Jung; Burns, Timothy P.; Uhm, Sung T.
- CS Dep. Chem., Univ. Nebraska-Lincoln, Lincoln, NE, 68588, USA
- SO Journal of Organic Chemistry (1981), 46(21), 4323-4 CODEN: JOCEAH; ISSN: 0022-3263
- DT Journal

- LA English
- AB Highly reactive Zn and Mg metal powders can be prepd. by Li redn. of the corresponding metal salt with a catalytic amt. of naphthalene as an electron carrier. Applications to the Reformatskii reaction, the **Grignard reaction**, and cyclopropanation (with dibromomethane) are described.
- L16 ANSWER 60 OF 84 CAPLUS COPYRIGHT 2003 ACS on STN
- AN 1980:567250 CAPLUS
- DN 93:167250
- TI The photochemistry of 4-phenyl-1-iodobutane and 4-phenyl-2-iodomethyl-1-butene
- AU . Charlton, James Leslie; Williams, Gaynor Jane; Lypka, Gerald Nicholas
- CS Dep. Chem., Univ. Manitoba, Winnipeg, MB, R3T 2N2, Can.
- SO Canadian Journal of Chemistry (1980), 58(12), 1271-4 CODEN: CJCHAG; ISSN: 0008-4042
- DT Journal
- LA English
- AB The photochem. of 4-phenyl-1-iodobutane (I) and 4-phenyl-2-iodomethyl-1-butene (II) is examd. to det. the effect of structure on the character of the intermediates generated on photolysis. The irradn. of I gives only radical intermediates. By contrast the photolysis of II gives products that are more consistent with cationic intermediates.
- L16 ANSWER 61 OF 84 CAPLUS COPYRIGHT 2003 ACS on STN
- AN 1979:167959 CAPLUS
- DN 90:167959
- TI Direct halogen substitution of 1,2-halohydrins by organomagnesiums and lithiums in presence of copper salt
- AU Normant, J. F.; Mulamba, T.; Scott, F.; Alexakis, A.; Cahiez, G.
- CS Lab. Chim. Organoelements, Univ. Pierre et Marie Curie, Paris, Fr.
- SO Tetrahedron Letters (1978), (39), 3711-12 CODEN: TELEAY; ISSN: 0040-4039
- DT Journal
- LA French
- MeCHRCH2OMgCl (I; R = Cl, Br, iodo) reacted with BuMgCl, in the presence of 5% CuBr, by 3 routes: by intermediate epoxide formation giving MeCH(OH)CH2Bu (II) (2, 2, 52% resp.), by direct substitution giving MeCHBuCH2OH (III) (29, 44, 0%, resp.), and by Tiffeneau rearrangement giving EtCH(OH)Bu (IV) (14, 6, 0%, resp.). Vinylic and allylic organomagnesiums reacted selectively by direct substitution. Thus R1MgCl (V; R1 = CH2:CH, Ph, CH2:CHCH2, CH2:CMeCH2, Me2C:CHCH2) with I (R = Br) in the presence of 5% CuBr in THF followed by H3O+ gave 60-80% MeCHR1CH2OH. In the absence of CuBr, V (R1 = Me2C:CHCH2) reacted with I (R = Br) in THF at 20.degree. for 2 h to give 77% CH2:CHCMe2CHMeCH2OMgCl. The direct halogen substitution reaction is also applicable to organolithiums. Thus BuLi with MeCHBrCH2OLi in Et2O in the presence of 5% CuBr at 10.degree. for 2 h gave 2% II, 40% III, and 20% IV.
- L16 ANSWER 62 OF 84 CAPLUS COPYRIGHT 2003 ACS on STN
- AN 1978:614835 CAPLUS
- DN 89:214835
- TI Synthesis of acetylenic alcohols by alkylation of .omega.-hydroxy-1-alkynes
- AU Flahaut, Jacques; Miginiac, Philippe
- CS Lab. Chim. Organomet., Univ. Poitiers, Poitiers, Fr.
- SO Helvetica Chimica Acta (1978), 61(6), 2275-85 CODEN: HCACAV; ISSN: 0018-019X

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DT Journal
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LA French

AB HC.tplbond.CCHRCR1R2OH (R = R1 = R2 = H; R = R1 = H, R2 = alkyl; R = H, R1 and R2 are alkyl; R = Pr, R1 = R2 = Et), HC.tplbond.CCH2CH2CRR1OH (R = R1 = H; R = H, R1 = Me; R = Me, R1 = Et), and HC.tplbond.C(CH2)3CHROH (R = H, Pr) were alkylated by alkyl bromides and iodides and LiNH2 to give R3C.tplbond.CCHRCR1R2OH, R3C.tplbond.CCH2CRR1OH, and R3C.tplbond.(CH2)3CHROH (R3 = Me, Et, Bu, Pr).

L16 ANSWER 63 OF 84 CAPLUS COPYRIGHT 2003 ACS on STN

AN 1977:71780 CAPLUS

DN 86:71780

- TI Study of the reaction of organometallic compounds (M = zinc, magnesium, lithium) with conjugated enymes. I. The hydrocarbons HC.tplbond.C-C(R):C(R')(R'"); effect of the structure on reactivity and regionelectivity
- AU Mesnard, D.; Miginiac, L.
- CS Lab. Synth. Org., Univ. Poitiers, Poitiers, Fr.
- SO Journal of Organometallic Chemistry (1976), 117(2), 99-115 CODEN: JORCAI; ISSN: 0022-328X
- DT Journal
- LA French
- AB Reactive organometallic compds. such as allylzinc, -magnesium, lithium and satd. lithium compds. readily undergo addn.
 reactions with conjugated enymes., HC.tplbond.CCR:CR1R2, but the
 reactivity is reduced when the steric hindrance around the double bond is
 increased. With each organometallic compd. used, this reaction is
 regioselective: 3,4-addn. with organozinc compds., 1,2-addn. with
 organolithium compds. (allyl, butyl), and both 1,2- and 1,4-addn. with
 organomagnesium compds.
- L16 ANSWER 64 OF 84 CAPLUS COPYRIGHT 2003 ACS on STN
- AN 1976:170295 CAPLUS
- DN 84:170295
- TI Catalyst
- IN Carney, Robert L.
- PA Zoecon Corp., USA
- SO U.S., 4 pp. CODEN: USXXAM
- DT Patent
- LA English
- FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
ΡI	US 3948803	Α	19760406	US 1974-473628	19740528
				US 1974-473628	19740528

- AB Organometallic catalysts are described for the alkylation of a Grignard reagent by an org. halide or an org. sulfonate ester. The catalyst is a soln. of a compd. of the formula MX(CuCr)n, where M is Li or Mg, X is chloro or bromo, and n is 1, 2, or 3, in a solvent. The preferred catalyst is Li[CuCl(CN)] in tetrahydrofuran. Other catalysts were prepd. from CuCN and LiBr or MgCl2. In an example, the catalyst was used to prep. Z-9-tricosene from amylmagnesium bromide Grignard reagent and Z-9-octadecenyl bromide.
- L16 ANSWER 65 OF 84 CAPLUS COPYRIGHT 2003 ACS on STN
- AN 1975:578229 CAPLUS
- DN 83:178229

- TI Synthesis of 3Z-nonenal and 3Z,6Z-nonadienal
- AU Kajiwara, Tadahiko; Odake, Yoshinobu; Hatanaka, Akikazu
- CS Dep. Agric. Chem., Univ. Yamaguchi, Yamaguchi, Japan
- SO Agricultural and Biological Chemistry (1975), 39(8), 1617-21 CODEN: ABCHA6; ISSN: 0002-1369
- DT Journal
- LA English
- AB 3Z-Nonenal and 3Z,6Z-nonadienal, potential biosynthetic precursors of 2E-nonenal and 2E,6Z-nonadienal, were for the first time synthesized stereoselectively.
- L16 ANSWER 66 OF 84 CAPLUS COPYRIGHT 2003 ACS on STN
- AN 1975:459799 CAPLUS
- DN 83:59799
- TI Aromatic hydrocarbon polymers
- IN Hay, Allan S.; Relles, Howard M.
- PA General Electric Co.
- SO U.S., 3 pp. CODEN: USXXAM
- DT Patent
- LA English
- FAN.CNT 1

ΡI

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
		-		
US 3810879	Α	19740514	US 1972-240786	19720403
			US 1972-240786	19720403

- AB Arom. diolefins, such as 2,2-bis(4-phenyl-3-cyclohexen-1-yl)propane (I), were manufd. by **Grignard reaction**, and polymd. in the presence of Li to give arom. hydrocarbon polymers, such as poly[2,2-bis(4-phenyl-3-cyclohexen-1-yl)propane] (II) useful for dielectrics. Thus, a mixt. of I 10 (by reaction of 2,2-bis(4-oxocyclohexyl)propane with bromophenyl Mg) and Li 6 in THF 200 parts was stirred for 11 hr at .apprx.25.degree. in N, and treated with 510 parts CH3OH to ppt. II with 90% yield and 26,500 mol. wt.
- L16 ANSWER 67 OF 84 CAPLUS COPYRIGHT 2003 ACS on STN
- AN 1975:443413 CAPLUS
- DN 83:43413
- TI Halomethyl-metal compounds. LXXIII. Grignard reagents derived from gem-dibromocyclopropanes. .alpha.-Bromocyclopropyltin compounds as precursors for .alpha.-bromocyclopropyllithium reagents by transmetalation
- AU Seyferth, Dietmar; Lambert, Robert L., Jr.
- CS Dep. Chem., Massachusetts Inst. Technol., Cambridge, MA, USA
- SO Journal of Organometallic Chemistry (1975), 88(3), 287-301 CODEN: JORCAI; ISSN: 0022-328X
- DT Journal
- LA English
- AB The reaction of Me2CHHgCl in THF with gem-dibromocyclopropanes give .alpha.-bromocyclopropylmagnesium chlorides which are unstable at room temp. and give carbene-derived products. At about -70.degree. these reagents are stable and can be used in synthesis. Protolysis gives a mixt. of syn and anti isomers when these are possible, but when treated with Me3SnCl, only the isomer with the Me2Sn substituent in the anti position is obtained. Treatment of syn-7-bromo-anti-7-trimethylstannylnorcarane with BuLi at -95.degree. gave only syn-7-bromo-anti-7-lithionorcarane; stereospecific reactions of this reagent with CO2 and C2Cl6 are described. In situ Grignard-Wurtz reactions were used to prepare 7,7-bis(trimethylsilyl)- and

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7,7-bis(trimethylstannyl)norcarane.

- L16 ANSWER 68 OF 84 CAPLUS COPYRIGHT 2003 ACS on STN
- AN 1974:551268 CAPLUS
- DN 81:151268
- TI Quasi-Favorskii rearrangement. Synthesis of 1-phenylcycloalkanecarboxylic acids
- AU Stevans, Calvin L.; Pillai, P. Madhavan; Taylor, K. Grant
- CS Dep. Chem., Wayne State Univ., Detroit, MI, USA
- SO Journal of Organic Chemistry (1974), 39(21), 3158-61 CODEN: JOCEAH, ISSN: 0022-3263
- DT Journal
- LA English
- AB .alpha.-Halo ketones without an .alpha.-H undergo C skeleton rearrangements on treatment with the Li salt of arom. primary amines in ether, yielding amides. The action of Li anilide on 1-benzoyl-1-bromocyclohexane gave 55% 1-phenyl-cyclohexanecarboxanilide and 30% 1-benzoyl-1-(phenylamino)-cyclohexane. The yield of the rearranged amide was improved by using a more hindered amine such as .omicron.-toluidine or 2,6-dimethylaniline. Also, a p-MeO substituent on the phenyl group of the .alpha.-halo ketone facilitated the rearrangement while a m-Cl substituent decreased the yield of the rearranged amide. The extension of this rearrangement to .alpha.-bromocycloalkyl Ph ketones and hydrolysis of the resulting amides gave 1-phenylcycloalkane-carboxylic acids. Treatment of endo-2-benzoyl-exo-2-bromonorborane with Li anilide gave endo-2-phenylnorbornane-exo-2-carboxanilide, as expected from a concerted semibenzilic rearrangement mechanism.
- L16 ANSWER 69 OF 84 CAPLUS COPYRIGHT 2003 ACS on STN
- AN 1973:477674 CAPLUS
- DN 79:77674
- TI Organometallic compounds. LIII. Mechanism of hydrolysis of organoaluminumate complexes
- AU Lehmkuhl, Herbert; Nehl, Hans
- CS Max-Planck-Inst. Kohlenforsch., Muelheim/Ruhr, Fed. Rep. Ger.
- SO Justus Liebigs Annalen der Chemie (1973), (4), 659-65 CODEN: JLACBF; ISSN: 0075-4617
- DT Journal
- LA German
- GI For diagram(s), see printed CA Issue.
- AB Competitive action of 1:1 H2O-D2O mixts. on the complex I showed that protolysis of I is preceded by rate-detg. complex formation of water with the Al compd. Hydrolysis of the Mg compd. II, of Al-alkyl (alkyl = Me or Et) bonds, and of Al-C bonds of other trialkylaluminum compds. revealed an isotope effect whose magnitude indicated that cleavage of the OH(D) bond detd. the rate of reaction.
- L16 ANSWER 70 OF 84 CAPLUS COPYRIGHT 2003 ACS on STN
- AN 1973:454020 CAPLUS
- DN 79:54020
- TI Preparation of poly(p-(.omega.-lithium alkyl)styrenes) and their use as polymer metalating agents
- AU Hallensleben, Manfred L.
- CS Inst. Makromol. Chem., Univ. Freiburg, Freiburg/Br., Fed. Rep. Ger.
- SO Angewandte Makromolekulare Chemie (1973), 31, 147-59 CODEN: ANMCBO; ISSN: 0003-3146
- DT Journal
- LA German

- AB The lithiated polymers I (n = 1-4) were prepd. by reaction of the corresponding halogen compds. with BuLi. I were as reactive as BuLi in the metalation of compds. such as fluorene [86-73-7] and 1-bromonaphthalene [90-11-9], and could be easily regenerated. The no. of active sites gradually decreased, owing to Wurtz-Fittig reactions between I and substrate.
- L16 ANSWER 71 OF 84 CAPLUS COPYRIGHT 2003 ACS on STN
- AN 1968:506783 CAPLUS
- DN 69:106783
- TI Reaction of organolithium and organomagnesium compounds with carbonyl derivatives of barenes and neobarenes
- AU L'vov, A. I.; Zakharkin, L. I.
- CS Inst. Elementoorg. Soedin., Moscow, USSR
- SO Izvestiya Akademii Nauk SSSR, Seriya Khimicheskaya (1967), (12), 2653-62 CODEN: IASKA6; ISSN: 0002-3353
- DT Journal
- LA · Russian
- GI For diagram(s), see printed CA Issue.
- Tertiary carboranyl alcs. are cleaved at C-C bond by catalytic amts. of AB RONa. Reaction of MeMgI with 0.3 mole 1-methyl-2-acetylcarborane (I) in Et20 gave 53% starting ketone, 42% methylcarboranyl(dimethyl)carbinol (II) and 4.8% methylcarborane (III); MeLi similarly gave 84% starting ketone and 16% III; similar reaction in tetrahydrofuran (THF) gave from MeMgI 43% starting ketone, 32% III and 25% II, while with MeLi 90% III was formed and 10% unreacted ketone was recovered. Methylcarboranylmagnesium bromide (IIIa) in THF treated with Me2CO 3 hrs. gave 8% II and 92% III. II kept overnight with EtONa in EtOH gave no residual II and a considerable amt. of III. MeMgI and methylcarboranylcarboxylyl chloride in Et20 gave 30% II, m. 87-9.degree.. EtMgBr and I in Et2O gave in 0.5 hr. 94% 1-methylcarboranylethanol, m. 153-4.degree.. 1-Methyl-7acetylneocarborane and MeMgI in Et20 0.5 hr. gave 91.5% starting ketone and 8.5% methylneocarboranyldimethylcarbinol (IV); MeLi similarly gave 55% above carbinol and 3% methylneocarborane, besides 42% initial ketone. Methylneocarboranyllithium (V) in C6H6 gave with Me2CO (20% excess) in 2 hrs. 95% IV, m. 29-30.degree.. MeMgI and 1-methyl-7-benzoylneocarborane gave in 2 hrs. in Et2O 62% (methylneocarboranyl)phenylmethylcarbinol, b1.cntdot.5 152-3.degree., n20D 1.5807, also obtained from AcPh and V. Similar reaction with PhMgBr gave 88% (methylneocarboranyl)diphenylcarbino 1, m. 90-1.degree.. MeMgI and 2,3-carborano-4,5-benzocyclopentanone in Et2O gave 82% VI m. 140-1.degree.; MeLi gave 76% VI, while PhLi gave 77% VII, m. 165-6.degree.. Bis(phenylcarboranyl) ketone and MeMgI in Et20 gave 82% bis(phenylcarboranyl)carbinol, m. 272-4.degree., while PhMgBr gave in 10 hrs. at 45.degree. in Et2O-C6H6 66.7% same carbinol. 1-Phenyl-2-benzoylcarborane (VIII) and MeMgI gave 45% (phenylcarboranylmethyl) methylcarbinol, m. 128-9.degree., while the filtrate from this was treated with CrO3 to yield VIII, phenyl(phenylcarboranyl)carbinol (IX) and (phenylcarboranyl)phenylmethylca rbinol, all isolated as acetates after acetylation with Ac2O. PhMgBr and VIII in THF gave 88% IX m. 122-4.degree., and some phenylcarborane (X); similar reaction in Et2O gave Ph2, X and Ph3COH as well as [o-PhCB10H10CC(OH)Ph]2, m. 239-40.degree.. PhLi and methylcarboranecarboxaldehyde (XI) in Et20 0.5 hr. gave 85% (methylcarboranyl)phenylcarbinol (XII), m. 107.degree.; the same formed from this reaction in THF. IIIa and XIa in Et20 gave 93.5% 1-(methylneocarboranyl)-2-carboranyl-1-ethanol, m. 239-40.degree... appropriate aldehyde and methylcarboranyllithium gave 95% bis(methylcarboranyl)carbinol, m. 177-8.degree.. XII and EtONa-EtOH in 12

hrs. at 20.degree. gave III as the sole product. iso-PrMgBr and XI in Et20 gave 92% methylcarboranylcarbinol, m. 268-9.degree. Ozonolysis of vinyl or allyl carborane or neocarborane and treatment of the mixt. with Me2S at -30.degree. gave after warming and an aq. treatment the following aldehydes RCHO: o-HCB10H10C, m. 212-13.degree. (2,4dinitrophenylhydrazone, m. 183-4.degree.); o-MeCB10H10C, m. 220-2.degree. (189.degree.); o-HCB10H10CCH2, m. 94-5.degree. (m. 163-4.degree.); o-MeCB10H10CCH2, m. 140-2.degree. (m. 176-7.degree.); m-MeCB10H10C, m. 143-4.degree. (m. 177-8.degree.); m-MeCB10H10CCH2, b2 98.degree., (m. 154-5.degree.). Carboranyllithium or corresponding RMgX in THF treated with ethoxymethyleneaniline and heated 1 hr. gave after an aq. treatment and steam distn. of the org. layer with dil. H2SO4 40-55% carboranecarboxaldehyde, XI and 25% XIa. (Methylneocarboranyl)methylcarbi nol acetate, b7 127-30.degree., gave 1-methyl-7-vinylneocarborane, m. 75-6.degree.. Allyl bromide added to V in C6H6 and heated 2 hrs. gave 80% 1-methyl-7-allylneocarborane, b7 95-6.degree., n22D 1.5260, d20 0.9040. Similarly was prepd. 80% 1-methyl-2-allylcarborane, b13 142-3.degree., d20 0.9295.

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L16 ANSWER 72 OF 84 CAPLUS COPYRIGHT 2003 ACS on STN
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AN 1964:483858 CAPLUS

DN 61:83858

OREF 61:14557b-d

- TI The action of anhydrous cobalt **chloride** and anhydrous uranyl nitrate on several aromatic organomagnesiums and organolithiums. Coupling reactions. I
- AU Morizur, Jean Pierre
- CS Ecole Natl. Super. Chim., Paris
- SO Bulletin de la Societe Chimique de France (1964), (6), 1331-7 CODEN: BSCFAS; ISSN: 0037-8968
- DT Journal
- LA Unavailable
- AB The action of small amts. of anhyd. COCl2 on various Grignard reagents (RMgBr) in the presence of PhBr or BuBr gave the dimeric species R2. Compds. prepd. in this way were 55% 2,2'-dimethylbibenzyl; 53% 2,3-dibenzylbutane, b0.07 110.degree.; 55% 2,5-diphenylhexane; 50% 4,4'-diisopropylbiphenyl (II), 60% 4,4'-di(tert-butyl)biphenyl (III); 55% 1,1,2,2-tetraphenylethane (IV) 2,2'-dimethoxy-5,5'-dimethylbiphenyl (V), and 25% 2,2'bithiophene (VI). 4-H2C:CHCH2C6H4MqBr gave an uncharacterized polymeric product. Neither PhMgBr nor 4-BrC6H4MgBr gave the desired dimer. The latter gave 4-BrC6H4Bu with BuBr. Analogous dimerization . reactions were observed with various organolithium (RLi) compds. and COC12 in the presence of PhBr. Compds. prepd. in this way were 65% 2,2'-dimethylbiphenyl, 60% 4,4'-dimethylbiphenyl, 60% I, 60% II, 60% III, 60% IV, 55% 4,4'-dimethoxybiphenyl, 65% V, 65% 4,4'-(N,Ndimethylamino)biphenyl, and 30% VI. All of the coupling reactions are vigorously exothermic. Mechanisms for the reactions are suggested. The reaction of anhyd. UO2(NO3)2 with PhMgBr or PhLi proceeds vigorously to give C6H6, Ph2 (.apprx.25%), and a ppt. of UO3.x-H2O where x = 0.5-2.0.

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L16 ANSWER 73 OF 84 CAPLUS COPYRIGHT 2003 ACS on STN
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AN 1964:60729 CAPLUS

DN 60:60729

OREF 60:10625c-d

- TI Halogen-metal interchange reactions of 3,3,3-trichloro-1,2- epoxypropane and of chloral with organolithium compounds and Grignard reagents
- AU Reeve, Wilkins; Fine, Leonard W.
- CS Univ. of Maryland, College Park

- SO Journal of the American Chemical Society (1964), 86(5), 880-2 CODEN: JACSAT; ISSN: 0002-7863
- DT Journal
- LA Unavailable
- AB 3,3,3-Trichloro-1,2-epoxypropane reacts with MeLi or PhLi to form 3,3-dichloroallyl alc. and Me Cl or PhCl. The formation of these products shows that a halogen-metal interchange reaction has occurred. None of the products expected from the opening of the epoxide ring by a carbanion could be detected. Even with BuMgCl and with Bu2Mg, a similar halogen-metal interchange reaction occurs. This is believed to be the first case reported in which a Grignard reagent undergoes a halogen-metal interchange reaction rapidly and to the exclusion of the usually observed reaction path. With BuMgCl, the epoxide ring is also attacked by chloride ion, but not by the Bu carbanion, and the chlorohydrin is formed. PhLi also undergoes a halogen-metal interchange reaction with chloral rather than adding to the carbonyl group in the expected manner. The above reactions indicate that the Cl3C group is unusually reactive in the halogen-metal interchange reaction.
- L16 ANSWER 74 OF 84 CAPLUS COPYRIGHT 2003 ACS on STN
- AN 1963:422397 CAPLUS
- DN 59:22397
- OREF 59:4104e-g
- TI Optical rotatory properties of polyaldehydes
- AU Abe, Akihiro; Goodman, Murray
- CS Polytech. Inst. of Brooklyn, Brooklyn, NY
- SO Journal of Polymer Science (1963), Pt. A 1(6), 2193-205 CODEN: JPSCAU; ISSN: 0022-3832
- DT Journal
- LA Unavailable
- The optical rotatory activity of poly-(R)(+)-citronellal, poly-(R)(+)-6-methoxy-4-methylhexanal, and <math>poly-(S)(+)-2-methylbutanal are essentially independent of their intrinsic viscosity and polymer crystallinity. Changes in temp. alter the equils. among conformation forms. Enhancement of the optical activity arises from a conformational rigidity around the asymmetric center in the side chain of the polymer. (R)(+)-Citronellal (I) is obtained by vacuum distn., and the fraction b2 60-2.degree., [.alpha.]26D + 12.9.degree. (c 0.0558, C6H6) is collected. I is reduced to (R)(+)-citronellol in ether with LiAlH4, [.alpha.]23D + 4.53.degree. (c 0.0367, C6H6) and converted to the (R)(+)-citronellyl methyl ether (II), b1 58-60.degree., [.alpha.]23D 5.59.degree., (c 0.0838, C6H6). II is cleaved by ozonolysis and the ozonide reduced to (R)(+)-6-methoxy-4-methylhexanal (III), b2 45.degree., [.alpha.]26D + 2.47.degree. (c 0.0263, C6H6), n23D = 1.4301, d23D = 0.907. (R)(S)-2-Methylbutanal (IV), b.p. 92-5.degree. at 1 atm. is prepd. from 2-bromobutane by the Grignard reaction, and (S)(+)-2-methyl-butanal (V), [.alpha.]26.6D 30.7.degree. (c 0.0654, C6H6) is prepd. by the oxidn. of active primary amyl alc. Polymerizations of I, III, IV, and V in Et2O or hexane as solvent and with a variety of catalysts are described.
- L16 ANSWER 75 OF 84 CAPLUS COPYRIGHT 2003 ACS on STN
- AN 1963:3349 CAPLUS
- DN 58:3349
- OREF 58:541g-h
- TI The addition of methyl Grignard to 4-tert-butylcyclohexanone
- AU Houlihan, William J.
- CS Universal Oil Prod., East Rutherford, NJ

- SO Journal of Organic Chemistry (1962), 27, 3860-4 CODEN: JOCEAH; ISSN: 0022-3263
- DT Journal
- LA Unavailable
- The addn. of methyl Grignard to 4-tert-butylcyclohexanone was studied under a variety of exptl. conditions. It was found that the cis-trans ratio of the resultant 1-methyl-4-tert-butylcyclohexanol is affected by the methyl halide used to form the Grignard reagent, the solvent, and the addn. of magnesium halide. The magnesium source, presence of air, or cuprous halide had negligible effect on the reaction. For the reactions carried out in diethyl ether, the cis-trans ratios are methytmagnesium iodide--1.02, methylmagnesium bromide--1.36, dimethylmagnesium--1.40, methylmagnesium chloride--1.44, methylmagnesium bromide di(magnesium bromide)--1.84. The reaction of methylmagnesium bromide in various solvents gave anisole--1.11, diethyl ether--1.36, tetrahydrofuran--2.26. Methyllithium in diethyl ether gave a ratio of 1.86.
- L16 ANSWER 76 OF 84 CAPLUS COPYRIGHT 2003 ACS on STN
- AN 1962:403839 CAPLUS
- DN 57:3839
- OREF 57:728d-i,729a-q
- TI Reactions of .alpha.-dimethylaminophenylacetonitrile and its ethylation product with basic or nucleophilic reagents
- AU Morris, Gene F.; Hauser, Charles R.
- CS Duke Univ., Durham, NC
- SO Journal of Organic Chemistry (1962), 27, 465-71 CODEN: JOCEAH; ISSN: 0022-3263
- DT Journal
- LA Unavailable
- A study was made of the reactions of .alpha.-(dimethylamino)phenylacetonit AR rile (I) and its ethylation product (II) with basic or nucleo philic reagents including KNH2, BuLi, Grignard reagents, LiAlH4, and Na. reactions of I involved ionization of the .alpha.-H, addn. to the nitrile C, and displacement of the nitrile group from the .alpha.-C; those of II occurred at the nitrile C and .alpha.-C. Some interesting comparisons were made be tween the reactions of phenylacetonitrile and I and between those of I and II. I, b0.8 76-8.degree., n25D 1.5116, was prepd. in 94% yield from BzH and the appropriate reagents. I converted into its carbanion by KNH2 in NH3 and this carbanion ethylated with EtBr using 0.8 mole each of the reactants in 800 ml. NH3 gave 137-40 g. II, b0.5 70-2.degree., n25D 1.5113. In an attempt to effect self-condensation of I, a mixt. of 0.10 mole I and 0.05 mole KNH2 in 400 ml. liquid NH3 stirred 8 hrs., replaced by Et20, stirred 14 hrs. at 25-30.degree., 4 ml. AcOH added, followed by H2O, and worked up gave 76% I. BuLi (100 ml.) added to 16.7 g. I in 250 ml. Et2O cooled in an ice bath, after 6 hrs. 13 g. EtBr added, left 1 hr. at room temp., then 250 ml. 1.5N HCl added, refluxed overnight, the 2 layers sepd., the aq. acid layer extd. with Et2O, dried, and the residue distd. gave 6 g. propiophenone, b4.2 77-8.degree., n25D 1.5232; 2,4-dinitrophenylhydrazone m. 194-5.degree.. The acid layer made alk. and extd. with Et20 gave 8.9 g. 1-dimethylamino-1-phenyl-2-pentanone (III), b0.3 95-8.degree., n27D 1.5038. Similar results were obtained in other expts. in which the temp. was varied from -80.degree. to 35.degree. and the ratio of propiophenone to III varied considerably. EtMgI (139 ml., 0.793N) added to 16 g. I in 100 ml. Et2O, the mixt. stirred 24 hrs. at room temp., and the product distd. gave 13.8 g. 1-dimethylamino-1phenylpropane (IV), b0.3 41-2.degree., n24D 1.5012; picrate m. 167.5-9.0.degree.. The reaction was carried out under several sets of

conditions and the following resuits obtained (mole- equivs. EtMgI, temp., time in hrs., % yield of IV, % recovered I given): 0.5, 20.degree., 14, 36, 45; 1.0, 20.degree., 24, 67,n -; 1.0, 35.degree., 2, 48, -; 1.0, 35.degree., 24, 79, 9; 1.1, 35.degree., 24, 85, 0. Recovered I was characterized by acid catalyzed hydrolysis to BzH. BuMgBr (175 ml., 0.677N) added to 17.25 g. I in 250 ml. Et20 at 0.degree., after 48 hrs. the mixt. worked up, and the product distd. gave 16.85 g. 1dimethylamino-1-phenylpentane, b0.95 77-8.degree., n18D 1.5002; picrate m. 159-60.degree.. PhMgBr (from 0.2 mole PhBr) in 200 ml. tetrahydrofuran treated with 0.1 mole I in 100 ml. tetrahydrofuran, the mixt. refluxed 5 hrs., decompd., and evapd. gave 22 g. benzhydryldimethylamine-HCl, m. 217-22.degree.. A sample of this salt was hydrolyzed to liberate the free amine, m. 68-70.degree.; MeI salt m. 172-4.degree.. Chlorobenzylmagnesium chloride (from p-chlorobenzyl chloride) treated with I gave 1-dimethylamino-1-phenyl-2-(4chlorophenyl)ethane, b0.57 129-30.degree., n25.5D 1.5644; picrate m. 1345.5.degree.. tert-Butyl magnesium chloride (195 ml., 0.662 N) dild. with 300 ml. Et2O, 16 g. I in 50 ml. Et2O added, after 10 min. 11 g. EtBr added slowly, stirred 1 hr., treated with 100 ml. 2N HCl, the aq. acid layer heated 16 hrs., cooled, extd. with Et20, dried, and evapd. gave 1.36 g. benzaldehyde 2,4-dinitrophenylhydrazone. No propiophenone was detected. The aq. acid soln. gave 11.1 g. benzyldimethylamine, b15 66-7.degree., n26D 1.4987; picrate m. 93-4.degree.. LiAlH4 (7.6 g.) in 200 ml. Et20 treated with 32 g. I in 100 ml. Et20 to maintain reflux (1260 ml. H evolved), after refluxing 24 hrs. 50 ml. $\mbox{H2O}$ added slowly, the ether sepd., dried, evapd., and the residue distd. gave 17.2 g. material, b0.6 80.degree.. The distillate warmed 24 hrs. with 3N HCl and Et2O gave 14.9 g. 2-dimethylamino-2-phenylethylamine, b0.5 65.degree., n28D 1.5235. I (16 g.) added to 4.6 g. Na in 300 ml. liquid NH3, after 10 min. 16 g. EtI in 100 ml. Et20 added, left 1 hr., decompd., evapd., and the residue distd. gave 7.1 g. benzyldimethylamine, b5 56-8.degree., and 5.05 g. II, b0.4 70-2.degree.; benzyldimethylamine picrate m. 93-4.degree.. In another expt., in which no EtI was used, was obtained 62% benzyldimethylamine. 2-Morpholino-2-phenylacetonitrile (V) (0.1 mole) in Et2O stirred 16 hrs. with 0.3 mole PhCH2MgCl and the product crystd. gave 20 g. 1-morpholino-1,3-diphenyl-2-propanone, m. 55-60.degree.; HCl salt m. 204-6.degree.. KNH2 (0.2 mole) in 500 ml. liquid NH3 treated 4 hrs. with 37.6 g. II, the product treated with 10.6 g. NH4Cl, evapd., and the product collected gave 22.6 g. 2-dimethylamino-2-phenylbutyramidine (VI), m. 126-7.degree. (C6H6-hexane). Cyclization was effected using 3 g. acetylacetone and 1 g. anhyd. K2CO3 to 6.1 g. VI in 50 ml. alc., refluxing 16 hrs., pouring into 400 ml. H2O, and crystg. to give 2.4 g. 2-(1-phenyl-1-dimethylaminopropyl)-4,6dimethylpyrimidine, m. 223-5.degree.(MeOH). When II was treated with LiNH2 no amidine was isolated and 91% II was recovered. BuLi (125 ml. 1.64N) treated dropwise with 36.2 g. II in 150 ml. Et2O, the soln. left overnight, cooled, treated with H2O, extd. with Et2O, and the residue distd. gave 26 g. 3-phenyl-3-dimethylamino-4-octane ketimine (VII), b0.27 114.degree., n24D 1.5218. VII (5.2 g.) heated 20 hrs. with 5% HCl at 50.degree. gave 4.4 g. 3-phenyl-3-dimethylamino4-octanone, b0.3 104-5.degree., n22D 1.5132. PhLi (500 ml. 0.48 N) refluxed 3 hrs. with 45 g. II in 100 ml. Et20, left overnight, and the product distd. gave 44.8 g. 2-dimethylamino-1,2diphenyl-1-butanone ketimine (VIII), b0.3 134-4.degree., m. 76.58.0.degree.. VIII (10 g.) hydrolyzed 16 hrs. with 5% HCl gave 93% 2-dimethylamino-1,2-diphenyl-1-butanone, b0.25 143-4.degree., n26D 1.5784. LiAlH4 (5.9 g.) in 200 ml. Et2O treated dropwise while refluxing with 28.2 g. II in 75 ml. Et20, stirred 7 hrs., and the mixt. worked up gave 22 g. 1-dimethylamino-1-phenylpropane, b0.7

43-4.degree., n23.5D 1.5010; picrate m. 167.5-9.0.degree.. Na (9.2 g.) in 500 ml. liquid NH3 treated with 27.6 g. II, the soln. treated with 100 ml. Me3COH, evapd., the residue treated with 100 ml. H2O and 110 ml. concd. HCl, evapd., cooled, the acid soln. made alk., the mixt. extd. with Et2O, dried, the solvent removed, and the residue distd. gave $28.9 \ g.$ IV.

L16 ANSWER 77 OF 84 CAPLUS COPYRIGHT 2003 ACS on STN

AN 1956:44254 CAPLUS

DN 50:44254

OREF 50:8446c-h

TI The deuterium isotope effect in the methanolysis of some organometallic compounds

AU Wiberg, Kenneth B.

CS Univ. of Washington, Seattle

SO Journal of the American Chemical Society (1955), 77, 5987-90 CODEN: JACSAT; ISSN: 0002-7863

DT Journal

LA Unavailable

AΒ The Bu, Ph, and PhCH2 Grignard and Li reagents react with MeOD faster or as fast as with ordinary MeOH. A similar isotope effect was noted in the reaction of MeCHNaCO2Me (I) with MeOH. The results are compared with the data available in the literature. The isotope effect of the neutralization of the C-anion of PhCH(OH)CN (II) also was detd., and the rate law for the benzoin condensation has been reconsidered based on this evidence. MeOH and D2O fractionated through a column gave MeOD. BuMgBr (0.1 mole) in 80 cc. Bu2O refluxed in a slow stream of N, the mixt. treated with 1 cc. MeOD in 20 cc. Bu2O and heated, and the resulting C4H10 swept with N into a Dry Ice-Me2CO trap gave C4H10 contg. 33.5 .+-. 0.2% D. BuMgBr (from 2.1 g. BuBr and 0.28 g. Mg) in 15 cc. Bu20, refluxed in a slow stream of N and then added slowly to 5 cc. MeOD contg. 33.5-4.5% D in 15 cc. Bu20 with stirring, and the resulting butane isolated in the usual manner gave in 3 identical runs butane with 38.8, 38.9, and 36.3% D (isotope effect kH/kD 0.83, 0.83, and 0.87), resp. A similar reaction with an equiv. amt. Li for the Mg gave quite erratic results and low yields of butane which were overcome by using C6H6 as the solvent. BuBr gave thus with Li and MeOD contg. 33.5% D in 2 runs butane contg. 33.6 and 33.7% D (isotope effect 1.00 and 0.99), resp. PhMgBr from 3.1 g. PhBr, 0.50 Mg, and 15 cc. dry Et2O added with stirring to 8 cc. MeOD contg. 33.5 and 36.7% D, the mixt. treated after 10 min. with dil. HCl, and the org. layer dried and distd. gave C6H6, b. 78-82.degree., contg. 34.4 and 37.5% D, resp. (isotope effect 0.96, 0.97); the C6H6 contg. small amts. of Et2O and Bu20 which did not interfere with the mass spectrum analysis. and MeOH contg. 34.5 and 33.5% D gave C6H6 contg. 37.1 and 35.7% D (isotope effect 0.89 and 0.91), resp. PhCH2MgCl from 2.5 g. PhCH2Cl and 0.50 g. Mg added to MeOD contg. 33.5 (36.7)% D, and the mixt. worked up in the usual manner gave toluene contg. 35.0 (36.9)% D [isotope ratio 0.93 (0.99.)]. EtCO2Me (3 cc.) added with shaking to 100 cc. 0.45N Ph3CNa, the resulting Et2O soln. of I added with stirring to 15 cc. MeOD contg. 75.7% D, the mixt. treated after 0.5 min. with stirring rapidly with 55 cc. N HCl, and the Et2O soln. washed, dried, and distd. gave 1.5 cc. EtCO2Me, b. 77-9.degree., contg. 72.8% D (isotope effect 1.16). The benzoin condensation carried out as described previously (C.A. 49, 13184e) but with 93.7% pure EtOD showed after 75 and 120 min. 23 and 32% exchange with rate consts. ke of 3.5 and 3.2 .times. 10-3, resp.; with 52.0% pure EtOD 10 and 16% exchange occurred after 75 and 120 min., resp., with an av. rate const. ke of 1.5 .+-. 0.1 .times. 10-3.

L16 ANSWER 78 OF 84 CAPLUS COPYRIGHT 2003 ACS on STN

AN 1956:35956 CAPLUS

DN 50:35956

OREF 50:7072d-g

TI Addition reactions of triazenes, illustrating the reactivity of N:N double bonds

AU Klages, Friedrich; Mesch, Walter

CS Univ. Munich, Germany

SO Chemische Berichte (1955), 88, 388-96 CODEN: CHBEAM; ISSN: 0009-2940

DT Journal

LA Unavailable

AB When azo compds. are converted to triazenes, the N:N double bonds become less ready to undergo nucleophilic addn. reactions so that triazene derivs. cannot be obtained by such reactions (cf. Gilman and Pickens, C.A. 19, 2936). Reaction of PhN:NNHPh (I) with EtMgBr in Et2O or tetrahydrofuran leads to elimination of C2H6 and recovery of I when the red complex is hydrolyzed with dil. aq. NH4Cl. I is also recovered when the red complex of I with PhLi in Et20 or C6H6 solns. is destroyed, or on decompn. of the complex of I with LiAlH4, m. 245.degree., in Et2O or tetrahydrofuran. Reaction of I with EtMqBr in Et20 and refluxing with BzCl leads to formation of N; on hydrolysis PhNBz2 (II), m. 161.degree., is formed, while the same reaction at 0.degree. gives PhN2Cl (III) as well as II. Reaction of EtMgBr in Et20 with PhN:NHMe, then with BzCl, gives C2H6, N2, and II. PhN:NNBzPh with BzCl in Et2O in the presence of SbCl5, BF3, ZnCl2, or MgBr2 with cooling yields III after hydrolysis. EtMgBr with PhN:NNMe2 (IV) in tetrahydrofuran gives C2H6, N, and, after hydrolysis of the complex, 1,4-diphenyl-2,5-diethylhexahydro-1,2,4,5tetrazine, m. 124.degree., as well as MeNH2. An addn. mechanism.is proposed for its formation, which is accompanied by a secondary reaction in which the N is eliminated. IV with dry LiAlH4 at 120.degree. gives PhNH2 and NHMe2, this reaction can be regarded as reduction with decompn. of the N chain. p-MeC6H4N:NNMe2 (from PhNMe2 and p-MeC6H4N2X), m. 51.degree., with PhLi in Et2O forms N and m-MeC6H4CH2NHMe, characterized as the H oxalate, m. 205.degree., and as the p-toluenesulfonamide, m. 81.degree.; a mechanism is presented for this reaction.

L16 ANSWER 79 OF 84 CAPLUS COPYRIGHT 2003 ACS on STN

AN 1956:24279 CAPLUS

DN 50:24279

OREF 50:5008f-i

TI Some properties of esters of p-toluenesulfonic acid and 17.beta.-hydroxy sterols. III. Reaction of 17-tosylate of 5-androstene-3.beta.,17.beta.-diol and its 3-acetate with organomagnesium compounds and halogen salts of magnesium

AU Madaeva, O. S.

CS S. Ordzhonikidze All-Union Research Chem.-Pharm. Sci. Inst., Moscow

SO Zhurnal Obshchei Khimii (1955), 25, 1427-31 CODEN: ZOKHA4; ISSN: 0044-460X

DT Journal

LA Unavailable

AB cf. C.A. 47, 3326c. MeMgI, from 0.15 g. Mg, treated with cooling with 0.96 g. 17-tosylate of 5-androstene-3.beta.,17.beta.-diol, and refluxed 3 hrs. gave after usual aq. treatment 0.6 g. 17-iodo-5-androsten-3.beta.-ol, m. 135-5.5.degree. (from MeOH). Similar reaction with EtMgCl, completed in C6H6 at 60.degree. 4 hrs. gave a tarry product which was acetylated with Ac20-pyridine and the product was chromatographically purified on Al203 in petr. ether-C6H6, yielding retroandrostadienol 3-acetate, m. 95-5.5.degree.. Reaction of 5-androstene-3.beta.,17.beta.-diol 3-acetate

17-tosylate with MeMgI similarly gave 17-iodo-5-androsten-3.beta.-ol 3-acetate, m. 151-2.degree.. Cholesterol and p-MeC6H4SO2Cl in pyridine gave 82% cholesteryl tosylate, m. 131.degree.. This (0.1 g.) heated with 10 ml. Me2CO and 0.1 g. LiCl in a sealed tube 9 hrs. at 92-4.degree. gave .beta.-cholesteryl chloride, m. 95.degree.. Borneol and p-MeC6H4SO2Cl in pyridine gave borneol tosylate, m. 66-7.degree., which with MeMgI gave only a tarry material containing free iodine and free of S. The tosyl group in the 17-position is not replaced by the action of ZnI2 in Et2O, MgI2 or alkali metal iodides in Me2CO. Also in J. Gen. Chem. U.S.S.R. 25, 1373-6(1955) (Engl. translation).

L16 ANSWER 80 OF 84 CAPLUS COPYRIGHT 2003 ACS on STN

AN 1956:16132 CAPLUS

DN 50:16132

OREF 50:3300c-q

TI Reactions of the carbonamide group. III. Reaction with organometallic compounds

AU Heyns, Kurt; Pyrus, Wolfgang

CS Univ. Hamburg, Germany

SO Chemische Berichte (1955), 88, 678-83 CODEN: CHBEAM; ISSN: 0009-2940

DT Journal

LA Unavailable

cf. C.A. 48, 1261c. Amides, R'CONHR'' (including polypeptides), react AB with a 3- to 5-fold excess of RMgX or RLi, in suitable solvents and at sufficiently high temp., to produce, after hydrolysis, R'COR + H2NR''. AcNHMe, PhNHAc (I), Et hippurate, BzNHCH2CPh2OH (II), Et aceturate, Et N-glycylglycinate (III), H2NCH2CONHCH2CPh2OH (IV), or Me N-(N-leucylglycyl)glycinate (V) reacted thus. R in the RLi was Ph (at 36.degree.) or Et; reaction times for RLi were 1-3 hrs. R in RMgX was Ph (at 150.degree. in most cases), Me, Et, or Bu; times were 4-30 hrs. Yields of R'COR were only about 20-40%, and the reaction is therefore not practical for preparative cleavage of proteins. Caprolactam and BuMgBr at 150.degree. gave 2-butyl-.DELTA.1-hexamethylenimine 14.5% and 2,2-dibutylhexamethylenimine 6.7%, isolated as the picrolonates, m. 173.degree. and 118.degree., resp. H in -CONH- reacts with MeMgI in the Zerevitinov detn. if the detn. is performed in anisole at 120.degree.; compds. tested and moles of CH4 evolved were: I, 1.02 and 1.00; II, 2.01 and 2.02; AcNHCH2CPh2OH (VI), 2.01 and 1.99; III.HCl, 3.82 and 3.84; V.HCl, 4.78 and 4.88; IV, 4.71 and 4.78; N-glycylglycine, 0.02 and 0.01; 2,4-piperazinedione, 0.01 and 0.03. EtMgBr and I, heated 1 hr., then treated with AcCl or PrCOCl, gave PhNAc2 or PhNAcCOPr, but II or VI, treated in the same way, were dehydrated to 53% BzNHCH:CPh2, m. 131.degree., or 46% AcNHCH:CPh2 (VII), m. 161.degree., resp. VII was prepd. also from VI and Ac2O at 150.degree. in a sealed tube. VI, m. 138.degree., was prepd. from Et aceturate and PhMgBr. .alpha.-Aminoisocaprophenone-HCl, m. 199-203.degree. (decompn.) (2,4-dinitrophenylhydrazone-HCl, m. 202.degree.), was prepd. by reaction of concd. HCl at 135.degree. in a sealed tube with N-(1-benzoyl-3methylbutyl)benzamide (VIII), m. 107.degree.. VIII was obtained from N-benzoylleucyl chloride and AlCl3 in C6H6. .alpha.-Aminoacetophenone 2,4-dinitrophenylhydrazone-HCl m. 221.degree..

L16 ANSWER 81 OF 84 CAPLUS COPYRIGHT 2003 ACS on STN

AN 1955:73455 CAPLUS

DN 49:73455

OREF 49:13925f-q

TI Reactions between triphenyltin chloride and dilithium or

diGrignard compounds

- AU Zimmer, Hans; Mosle, H. G.
- CS Tech. Univ. Berlin-Charlottenburg
- SO Chemische Berichte (1954), 87, 1255-7 CODEN: CHBEAM; ISSN: 0009-2940
- DT Journal
- LA Unavailable
- The compds. formed are of the type (Ph3Sn)2R, rather than Ph3SnRLi(or MgBr), even though an excess of the Li or Grignard may be used. Ph3SnCl (19 g.) (in ether suspension) with an equiv. amt. of p-C6H4Li2 soln. (from p-C6H4Br2 and BuLi) gave 13 g. p-C6H4(SnPh3)2, m. 289-92.degree.. Ph3SnCl with 4,4'-dilithiobiphenyl gave an unstated yield of 4,4'-bis(triphenyltin)biphenyl, m. 235-6.degree.. Ph3SnCl with (BrMgCH2CH2)2 gave (Ph3SnCH2CH2)2, m. 149-50.5.degree..
- L16 ANSWER 82 OF 84 CAPLUS COPYRIGHT 2003 ACS on STN
- AN 1954:14291 CAPLUS
- DN 48:14291
- OREF 48:2575c-q
- TI Application of ultrasonic waves to the preparation of organometallic compounds
- AU Renaud, Pierre
- SO Bulletin de la Societe Chimique de France (1950) 1044-5 CODEN: BSCFAS; ISSN: 0037-8968
- DT Journal
- LA Unavailable
- The effect of ultrasonic waves on the prepn. of organometallic compds. of AΒ Li, Ca, Hg, Al, Be, and Zn is studied. They do not overcome the inertia of halogens in the formation of Grignard reagents. The prepn. of organo compds. of Li under the influence of ultrasonic waves takes place rapidly in Et2O even at 66.degree. B.acte.e. but is impossible in Bu2O. Derivs. of Ca cannot be obtained even by aid of EtMgI. Hg emulsifies immediately in Et20 but does not react with bromides. Al affords organo-Al compds. by reaction of an organo-Mg deriv. on Al powder; the use of Al-Mg alloy is unnecessary. Organo-Zn compds. are not obtained by an analogous reaction between RMgX and Zn in the presence of Cu. Beryllium does not resemble Al in its action. The halides of Fe, Ni, and Co give condensation reactions, catalyzed by a complex such as [CoCl2(NH3)4]Cl but not by [CoCl3(NH3)3]. In the production of Grignard compds. chlorides do not react, even with excitement by a bromide, except when Cl is mobile as in PhCH2Cl. CHCl3 and CCl4 retain their inhibiting action. Generally, only aliphatic or aromatic iodides and bromides react within a few min. or more slowly if a solvent (Et20, Bu20) is used. EtBr and PhBr do not attack Mg in the absence of a solvent. MeCH:CHBr does not furnish an organo-Mg deriv. even in presence of HgCl2. HC.tplbond.CHCH2Br only gives such a deriv. in the presence of HgCl2; the reaction is more rapid than that induced by simple heating. Mg 2-pyridyl bromide is obtained without heating but the further reaction with AcH is impossible without recourse to heat. EtMgBr, BuMgBr, and PhMgBr are obtained with the aid of ultrasonic waves in slightly aq. Et20 or Bu20, contg. up to 50% of C6H6, light petroleum, or even palm oil.
- L16 ANSWER 83 OF 84 CAPLUS COPYRIGHT 2003 ACS on STN
- AN 1941:47679 CAPLUS
- DN 35:47679
- OREF 35:7368a-f
- TI Factors determining the course and mechanisms of **Grignard**reactions IV. The effect of metallic halides on the reaction of
 al Grignard reagents and organic halides

- AU Kharasch, M. S.; Fields, E. K.
- SO Journal of the American Chemical Society (1941), 63, 2316-20 CODEN: JACSAT; ISSN: 0002-7863
- DT Journal
- LA Unavailable
- AB Co does not react with PhBr at 40.degree. after 1 hr. PhMgBr (I) gives 6-8% of Ph2 (II). I (0.14 mole) and 9 mol.% of CoCl2 (III) give 27% II; in the following the figure before the metal halide is mol.%; 0.54 mole I, 0.4 mole PhBr (IV) and 7 III give 83% II; 0.113 mole I, 0.1 mole IV and 2.5 III give 86% II. IV takes part in this reaction, as can be demonstrated by a halogen titration of the aq. soln. obtained by hydrolysis of the reaction mixt. after the reaction has ceased and by the fact that only a portion of IV can be recovered; thus, IV acts as an oxidizing agent in converting I into II. The II is formed exclusively from I because IV can be replaced by p-BrC6H4Me (with 9 III 86% II), EtBr (with 7 III 81% II) or iso-PrCl (with 5 III 58% II). The org. radical of IV is responsible for the formation of higher-boiling compds.; thus, 0.54 mole I, 0.4 mole II and 7 III in ether (heated 2 hrs.) give 18 g. C6H6, 34.5 g. Ph2, 1.7 g. terphenyl, 0.8 g. quaterphenyl and 17.5 g. very high-boiling material; such compds. are not found with aliphatic halides. PhCl (4 III) gives only 37% II. PhMgI and 28 III in ether-C6H6 at 0.degree. for 3 hrs. give 64% II; with 0.1 mole IV, 4 III gives 86% II. With I and IV other metal halides give the following results: 9 CuCl 6% II, 4 MnCl2 21% II, 5 FeCl3 47% II, 4 NiCl2 72% II, 4 CrCl3 7% II. Other reactions were also studied; thus, p-MeC6H4MgBr and IV with 10 III give 95% (p-MeC6H4)2; o-MeC6H4MgBr and EtBr with 7 III give 75% of (o-MeC6H4)2; p-MeOC6H4MgBr and EtBr with 5 III give 76% of (p-MeOC6H4)2; o-EtOC6H4MgBr and EtBr with 5 III give 74% of (o-EtOC6H4)2. It is believed that these reactions proceed through the agency of a Co subhalide, the active chain carrier; suggested reactions are: I + III .fwdarw. PhCoCl + MgBrCl; 2PhCoCl .fwdarw. Ph2 + 2CoCl; CoCl + PhBr .fwdarw. CoClBr + Ph-; x(Ph-) .fwdarw. C6H6, Ph2, C6H4Ph2, (6H4Ph)2, etc. These equations are used to explain the results obtained in part III. The data point to a definite relation between the electronegativity of the org. radical, the stability of the intermediate organometallic compd. and the rate of the normal addn.

L16 ANSWER 84 OF 84 CAPLUS COPYRIGHT 2003 ACS on STN

AN 1939:41261 CAPLUS

DN 33:41261

OREF 33:5804f-h

TI Synthesis of primary amines by the reaction of .alpha.-methylhydroxylamine with organomagnesium and organolithium compounds

AU Sheverdina, N. I.; Kocheshkov, K. A.

SO Zhurnal Obshchei Khimii (1938), 8, 1825-30 CODEN: ZOKHA4; ISSN: 0044-460X

DT Journal

LA Unavailable

AB RMgX and RLi in ether soln. at a temp. of -10.degree. to -15.degree. react readily with MeONH2 (I) to give primary amines. The yield depends on the nature of X, decreasing sharply from Cl to I, and is practically independent of the nature of R. The following compds. were reacted with I: EtMgBr, iso-AmMgCl, iso-AmMgBr, iso-AmMgI, sec-BuMgCl, tert-BuMgCl, PhMgCl, PhMgI, p-BrC6H4MgBr and PhLi. The yields of RNH2 were resp. 66.6, 80.1, 71.4, 5.3, 73.4, 73.6, 65.0, 0.23, 72.5 and 63.0%.

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(FILE 'HOME' ENTERED AT 16:14:11 ON 24 SEP 2003) FILE 'REGISTRY' ENTERED AT 16:14:32 ON 24 SEP 2003 Ll STRUCTURE UPLOADED 210 S L1 SSS FULL L2FILE 'CAPLUS' ENTERED AT 16:15:16 ON 24 SEP 2003 309 S L2 L3 L40 S L3 AND SYN THESIS AND PREPARATION L5 48 S L3 AND SYNTHESIS L6 . 3 S L5 AND LITHIUM L7 2 S L5 AND MAGNESIUM L8 194 S GRIGNARD REACTION AND LITHIUM AND MAGNESIUM 70 S L8 AND PHENYL L9 30 S L8 AND PHENYL AND CHLORIDE L10 L1110 S L8 AND PYRIDINE L12 1 S L8 AND PYRIMIDINE 0 S L8 AND PYRIDAZINE L13 L142 S L8 AND FURAN 1 S L8 AND THIEN L15. L16 84 S L8 AND CHLORIDE O S L16 AND PHENYL AND PYRIDINE AND FURAN AND THIEN AND PYRIMIDIN L170 S L8 AND PYRIDINE AND CHLIRIDE L181 S L8 AND PYRIMIDINE AND CHLORIDE L19 1 S L8 AND FURAN AND CHLORIDE L20 L21 1 S L8 AND THIEN AND CHLORIDE => d l18 fbib hitstr abs total L18 HAS NO ANSWERS 194 SEA FILE=CAPLUS PLU=ON GRIGNARD REACTION AND LITHIUM AND L8 MAGNESIUM O SEA FILE=CAPLUS PLU=ON L8 AND PYRIDINE AND CHLIRIDE L18 => d 19 fbib hitstr abs total ANSWER 1 OF 70 CAPLUS COPYRIGHT 2003 ACS on STN L9 ΑN 2002:882089 CAPLUS DN 137:384754 ΤI Preparation of exo-biperidens via the coupling of exo-silylenolethers with N-methylenepiperidinium salts ΙN Grosse, Markus; Klein, Peter; Thyes, Marco; Weber, Klaus Martin; Vilsmaier, Elmar PΑ Abbott Gmbh & Co. KG, Germany SO Ger. Offen., 12 pp. CODEN: GWXXBX DT Patent LΑ German FAN.CNT 1 PATENT NO. KIND DATE APPLICATION NO. DATE ----______ _____ DE 2001-10124453 20010518 PΙ DE 10124453 A1 20021121 A2 20021205 WO 2002-EP5497 20020517 WO 2002096874 WO 2002096874 A3 20030130 W: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NO, NZ, OM, PH,

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OS CASREACT 137:384754; MARPAT 137:384754

GI

III

AB A process for the prepn. of exo-biperidens I via the coupling of exo-silylenolethers II [R = alkyl, cycloalkyl] with N-methylenepiperidinium salts is disclosed. For example, coupling of exo-silylenolether II (R = Me), e.g., prepd. from cyclopentadiene and 3-buten-2-one in 2-steps, and N-methylenepiperidinium chloride, followed by the addn. of chlorophenylmagesium afforded a diastereomeric mixt. of exo-biperidens I. A key step of the process is the NaOMe mediated isomerization of endo-1-(bicyclo[2.2.1]hept-5-en-2-yl)ethan-2-one to the exo isomer.

- L9 ANSWER 2 OF 70 CAPLUS COPYRIGHT 2003 ACS on STN
- AN 1999:811200 CAPLUS
- DN 132:35338
- TI Process and catalysts for the symmetric disubstitution of carboxylic acid amides into substituted amines using Grignard reagents or organolithium compounds
- IN Buchholz, Herwig; Welz-Biermann, Urs; De Meijere, Armin
- PA Merck Patent GmbH, Germany
- SO PCT Int. Appl., 44 pp.
- CODEN: PIXXD2
- DT Patent

9/24/2003>

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AΒ
     Amides (e.g., diethylformamide) are sym. disubstituted on the geminal
     carbonyl-C atom of the amide using organolithium compds. or Grignard
     reagents (e.g., phenylmagnesium bromide) to give substituted amines [e.g.,
     (diphenylmethyl)diethylamine], which reaction is conducted in the presence .
     of a metal alcoholate (e.g., titanium tetraisopropoxide) catalyst and
     optional organosilane (e.g., tert-butylsilyl trichloride) cocatalyst.
RE.CNT 6
              THERE ARE 6 CITED REFERENCES AVAILABLE FOR THIS RECORD
              ALL CITATIONS AVAILABLE IN THE RE FORMAT
L9
     ANSWER 3 OF 70 CAPLUS COPYRIGHT 2003 ACS on STN
AN
     1999:811196 CAPLUS
DN
     Method and catalysts for the disubstitution of carboxylic acid amides with
TI
     two different organolithium or Grignard reagents in the preparation of
     substituted amines
     Buchholz, Herwig; Welz-Biermann, Urs
IN
     Merck Patent G.m.b.H., Germany
PΑ
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     PCT Int. Appl., 30 pp.
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    Amides (e.g., N-formylmorpholine) are disubstituted on the geminal
AΒ
     carbonyl-C atom of the amide using two different Grignard reagents (e.g.,
    phenylmagnesium bromide and methylmagnesium bromide) to give substituted
     amines [e.g., 1-[(1-phenyl)ethyl]morpholine], which reaction is
     conducted in the presence of a metal alcoholate (e.g.,
    tetraisopropoxytitanium) catalyst optionally with an organosilane (e.g.,
     chlorotrimethylsilane) cocatalyst.
              THERE ARE 9 CITED REFERENCES AVAILABLE FOR THIS RECORD
RE.CNT 9
              ALL CITATIONS AVAILABLE IN THE RE FORMAT
L9
    ANSWER 4 OF 70 CAPLUS COPYRIGHT 2003 ACS on STN
AN
     1999:811195 CAPLUS
DN
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     Catalytic titanium(IV) dioxide-induced geminal asymmetric disubstitution
ΤТ
     of carboxylic acid amides with organolithium or Grignard reagents for the
     preparation of substituted amines
     Buchholz, Herwig; Welz-Biermann, Urs
IN
PA · Merck Patent GmbH, Germany
SO
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	DE 19844194	A1 199	91223	DE 1998-19844194 19980926 DE 1998-19827167A119980618
	EP 1087931 R: AT, BE,			EP 1999-931109 19990618 GB, GR, IT, LI, LU, NL, SE, PT, FI DE 1998-19827167A 19980618 DE 1998-19844194A 19980926 WO 1999-EP4253 W 19990618
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FIRM	US 6479661	B1 200	21112	WO 1999-EP4256 W 19990618 US 2001-719971 20010323 DE 1998-19827167A 19980618 DE 1998-19844194A 19980926 WO 1999-EP4253 W 19990618
FAN	1999:811200 PATENT NO.	KIND DAT	E 	APPLICATION NO. DATE
PI	WO 9965862 W: JP, KR, RW: AT, BE, PT, SE	US		WO 1999-EP4254 19990618 FI, FR, GB, GR, IE, IT, LU, MC, NL,
	EP 1087933	A1 200	10404	DE 1998-19827161A 19980618 DE 1998-19827161 19980618 EP 1999-932702 19990618 GB, GR, IT, LI, LU, NL, SE, PT, FI DE 1998-19827161A 19980618 WO 1999-EP4254 W 19990618
	EP 1088029 R: AT, BE,		10404 , ES, FR,	
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	JP 2003524588	T2 200	30819	JP 2000-554208 19990618 DE 1998-19827161A 19980618 DE 1998-19827163A 19980618 DE 1998-19827164A 19980618 DE 1998-19827165A 19980618 DE 1998-19827166A 19980618 DE 1998-19827167A 19980618 DE 1998-19844194A 19980926

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WO 1999-EP4256 W 19990618
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        R: AT, BE, 'CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, PT, FI
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                                          DE 1998-19827165A 19980618
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                                          WO 1999-EP4257 W 19990618
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                                           WO 1999-EP4256 W 19990618
                            20030109
                                           US 2002-162257
                                                          20020605
    US 2003009029
                      A1
                                           DE 1998-19827164A 19980618
                                           US 2001-719810 A320010420
FAN
    1999:811202
     PATENT NO.
                     KIND
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PΙ
    WO 9965864
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                            19991223
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        W: JP, KR, US
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                                           DE 1998-19827167A 19980618
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                      А3
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PT, SE

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JP 2003524588
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                                      DE 1998-19844194A 19980926
                                      WO 1999-EP4256 W 19990618
US 2003009029
                       20030109
                                      ·US 2002-162257
                                                       20020605
                                      DE 1998-19827164A 19980618
                                      US 2001-719810 A320010420
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- OS CASREACT 132:35334; MARPAT 132:35334
- AB Amides (e.g., N-formylpiperidine) are disubstituted on the geminal carbonyl-C atom of the amide using two different Grignard reagents (e.g., phenylmagnesium bromide and ethylmagnesium bromide) to give substituted amines [e.g., 1-[(1-phenyl)propyl]piperidine], which reaction is conducted in the presence of a titanium dioxide catalyst and a metal alcoholate or organosilane (e.g., chlorotrimethylsilane) cocatalyst.
- RE.CNT 6 THERE ARE 6 CITED REFERENCES AVAILABLE FOR THIS RECORD ALL CITATIONS AVAILABLE IN THE RE FORMAT
- L9 ANSWER 5 OF 70 CAPLUS COPYRIGHT 2003 ACS on STN
- AN 1999:796156 CAPLUS
- DN 132:151849
- TI Grignard reagent formation from aryl halides. There is no aryl radical intermediate along the dominant reaction channel
- AU Garst, J. F.; Ronald Boone, J.; Webb, L.; Easton Lawrence, K.; Baxter, J. T.; Ungvary, F.
- CS Department of Chemistry, The University of Georgia, Athens, GA, USA
- SO Inorganica Chimica Acta (1999), 296(1), 52-66 CODEN: ICHAA3; ISSN: 0020-1693
- PB Elsevier Science S.A.
- DT Journal
- LA English
- AB For Grignard reagent formation from Mg and an aliph. halide RX in an ether solvent, a route through R.bul. is the major pathway. Part of the evidence is that byproducts of side reactions of R.bul. are formed in substantial yields. Similar reactions of Ph and o-(3-butenyl) phenyl halides give very low (sometimes trace) yields of byproducts derived from side reactions of R.bul., despite the fact that aryl R.bul. are much more reactive than alkyl in both solvent attack and cyclization [o-(3-butenyl)phenyl case]. Grignard reactions of aryl halides appear to proceed largely through a pathway along which R.bul. is not an intermediate. This is probably a dianion pathway, i.e., one along which RX2- is an intermediate or transition state.
- RE.CNT 16 THERE ARE 16 CITED REFERENCES AVAILABLE FOR THIS RECORD ALL CITATIONS AVAILABLE IN THE RE FORMAT

L9 ANSWER 6 OF 70 CAPLUS COPYRIGHT 2003 ACS on STN

AN 1999:407058 CAPLUS

DN .131:58656

TI Preparation of biphenyls having active substituents such as cyano group

IN Takahashi, Junya; Tsurushima, Masaaki

PA Mikuni Pharma Ind., Japan

SO Jpn. Kokai Tokkyo Koho, 9.pp.

CODEN: JKXXAF

DT Patent

LA Japanese

FAN.CNT 1

PATENT NO. KIND DATE APPLICATION NO. DATE

PI JP 11171799 A2 19990629 JP 1997-356313 19971208

JP 1997-356313 19971208

OS CASREACT 131:58656; MARPAT 131:58656

GΙ

$$\begin{array}{c|c} & & & \\ & & &$$

- AB Title compds. I [R = aryl, (aryl-contg.) hydrocarbyl; Z = active substituents; A = halo; k, m = 0, 1; k + m = 1; n = 0-2] are prepd. by reaction of R(C6H4)kMgX (R, k = same as I; X = halo) with aryl compds. II (Z, A, n, m = same as I; Y = leaving group) in the presence of Cu salts. 4'-Bromomethylbiphenyl-4-carbonitrile was condensed with BuMgCl in the presence of Li2CuCl4 in THF at room temp. overnight to give 89.5% 4'-pentylbiphenyl-4-carbonitrile with 98.9% purity.
- L9 ANSWER 7 OF 70 CAPLUS COPYRIGHT 2003 ACS on STN
- AN 1999:373858 CAPLUS
- DN 131:144631
- TI Preparation of heteroarylboron compounds
- AU Huang, Shi-Wen; Shan, Zi-Xing; Huang, Jin-Kun; Zhao, De-Jie
- CS College of Chemistry, Wuhan University, Wuhan, 430072, Peop. Rep. China
- SO Wuhan Daxue Xuebao, Ziran Kexueban (1999), 45(2), 160-164
- CODEN: WTHPDI; ISSN: 0253-9888
- PB Wuhan Daxue Xuebao Bianjibu
- DT Journal
- LA Chinese

AB 2-Thienyl boronic acid, di(2-thienyl)borinic acid, tri(2-thienyl)borane and their derivs. were prepd. by one-pot reaction between 2-bromothiophene, magnesium and boron trifluoride etherate or tri-Bu borate at room temp. 5-Methyl-2-furanyl and 2-benzofuranyl boron compds. were obtained from the corresponding lithium reagents reacting with boron trifluoride etherate or tri-Bu borate at lower temp.

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L9 ANSWER 8 OF 70 CAPLUS COPYRIGHT 2003 ACS on STN
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AN 1999:311426 CAPLUS

DN 130:338554

TI Preparation of substituted poly(arylenvinylenes) for use in electroluminescence

IN Spreitzer, Hubert; Kreuder, Willi; Becker, Heinrich; Schenk, Hermann; Yu,
Nu

PA Hoechst A.-G., Germany

SO Ger. Offen., 28 pp. CODEN: GWXXBX

DT Patent

LA German

FAN.CNT 1

ran.	CNII		
	PATENT NO.	KIND DATE	APPLICATION NO. DATE
PΙ			DE 1997-19748814 19971105
	CA 2308573	AA 19990520	CA 1998-2308573 19981022
			DE 1997-19748814A 19971105
			WO 1998-EP6722 W 19981022
	WO 9924526	A1 19990520	WO 1998-EP6722 19981022
	W: CA, CN,	JP, KR, MX, US	
	·		ES, FI, FR, GB, GR, IE, IT, LU, MC, NL,
	PT, SE	,,,	
	•		DE 1997-19748814A 19971105
	EP 1029019	A1 20000823	EP 1998-952737 19981022
		DE, FR, GB, IT,	
		, , , , ,	DE 1997-19748814A 19971105
			WO 1998-EP6722 W 19981022
	JP 2001522926	T2 20011120	JP 2000-520525 19981022
			DE 1997-19748814A 19971105
			WO 1998-EP6722 W 19981022
	US 2002064680	. A1 20020530	
	05 2002001000	. 111 20020330	DE 1997-19748814A 19971105
			WO 1998-EP6722 W 19981022
	HG 200200000	A1 20030508	
	05 2003088050	AI 20030508	
			DE 1997-19748814A 19971105
			WO 1998-EP6722 W 19981022

AB Poly(arylenevinylenes) bearing aryl substituents of specified structure, useful in electroluminescent lighting and displays, are prepd. Coupling di-Me bromoterephthalate with [3-[(3,7-dimethyloctyl)oxy]phenyl] boronic acid gave 98% di-Me 2-[3-[(3,7-dimethyloctyl)oxy]phenyl] terephthalate, redn. of which with LiAlH4 gave 82% corresponding bishydroxymethyl deriv., treatment of which with SOCl2 gave 70% 2,5-bis(chloromethyl)-3'-[(3,7-dimethyloctyl)oxy]biphenyl (I). Polymn. of 4.00 mmol each I and 2,5-bis(chloromethyl)-3'-[(3,7-dimethyloctyl)oxy]-4-methoxybiphenyl in dioxane contg. tert-BuOK at 99.degree. gave 44% copolymer with wt.-av. mol. wt. 1,350,000. Use of the products in LED's is exemplified.

L9 ANSWER 9 OF 70 CAPLUS COPYRIGHT 2003 ACS on STN

US 2000-530890 B120000822

AN 1996:739997 CAPLUS

DN 126:18658

TI Preparation of optically active biaryl compounds from 1-[2,6-(bistrifluoromethanesulfonyloxy)phenyl]naphthalene and Grignard reagents

IN Hayashi, Tamio

PA Sumitomo Chemical Co, Japan

SO Jpn. Kokai Tokkyo Koho, 6 pp.

CODEN: JKXXAF

DT Patent

LA Japanese

FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE		
ΡI	JP 08245551	A2	19960924	JP 1995-51091	19950310		
				JP 1995-51091	19950310		

OS CASREACT 126:18658; MARPAT 126:18658

GΙ

AB Optically active biaryl compds. I (R = alkyl, alkenyl, aryl, aralkyl) are prepd. by treatment of I (R = CF3SO3) with RMgX (R = same as above; X = halo) in the presence of optically active metal complex catalysts prepd. from transition metal compds. and optically active R2R3NCHR1CH2PR42 [R1-R3 = lower alkyl, aryl, aralkyl; R4 = (cyclo)alkyl, alkoxy, (halo-substituted) Ph; NR2R3 may form ring]. I (R = CF3SO3) was treated with PhMgBr, LiBr, and (S)-PhCH2CH(NMe2)CH2PPh2-Pd complex at 0.degree. for 48 h to give 74% (S)-I (R = Ph) (84% e.e).

- L9 ANSWER 10 OF 70 CAPLUS COPYRIGHT 2003 ACS on STN
- AN 1996:376669 CAPLUS
- DN 125:168189
- TI A regioselective addition reaction of a sulfonyl radical to conjugate enymesulfones: a convenient synthesis of 1,4-bis(arylsulfonyl)-1,3-butadiene
- AU Yoshimatsu, Mitsuhiro; Hayashi, Mitsumasa; Tanabe, Genzoh; Muraoka, Osamu
- CS Dep. Chem., Fac. Educ., Gifu Univ., Gifu, 501-11, Japan
- SO Tetrahedron Letters (1996), 37(24), 4161-4164 CODEN: TELEAY; ISSN: 0040-4039
- PB Elsevier
- DT Journal
- LA English
- OS CASREACT 125:168189

GΙ

AB P-MeC6H4SO2SePh regioselectively added to conjugate enynesulfones, e.g. (E)-HC.tplbond.CCH:CBrSO2Ph to give (1E,3E)-1,4-bis(arylsulfonyl)-1,3-butadienes, e.g. I (R = Br), which were converted to 4-heteroatom-substituted-1-phenylsulfonyl-1,3-butadienes, e.g. II. The stereochem. of PhCH2NHCH:C(SePh)CH:CH(SO2Ph), the PhCH2NH2 reaction product with I (R = H), was detd. by x-ray crystallog.

L9 ANSWER 11 OF 70 CAPLUS COPYRIGHT 2003 ACS on STN

AN 1996:264957 CAPLUS

DN 124:317470

TI Process for preparing silacyclohexane-based liquid crystal compounds

IN Kinsho, Takeshi; Shimizu, Takaaki; Ogihara, Tsutomu; Kaneko, Tatsushi; Nakashima, Mutsuo

PA Shin-Etsu Chemical Co., Ltd., Japan

SO Eur. Pat. Appl., 19 pp.

CODEN: EPXXDW

DT Patent

LA English

FAN.CNT 1

L'ETIA'	CIVI I				
	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
ΡI	EP 696591	A1	19960214	EP 1995-304845	19950711
	EP 696591	B1	20010314		
	R: DE, GB				
				JP 1994-182903 A	19940712
	JP 08081474	A2	19960326	JP 1995-198006	19950711
				JP 1994-182903 A	19940712
	US 5514824	Α	19960507	US 1995-501522	19950712
				JP 1994-182903 A	19940712
	EP 765880	A1	19970402	EP 1995-115429	19950929
	R: DE, GB				
		•		JP 1994-182903	19940712

OS CASREACT 124:317470; MARPAT 124:317470 GI

$$A_{R}$$
Si A_{I} A_{R} Si O_{II}

AB A process for prepg. compds. I (R, Y, A = org. residues; i = 0-3) via reacting a ketone compd. II (Ar = org. residue) with an organometallic reagent and subsequently dehydrating, oxidizing, and Ar removal to give I is described. Thus, Grignard reaction of 4-(4-pentyl-4-phenyl-4-silacyclohexyl)-3-cyclohexenone with trans-(4-propylcyclohexyl)phenylmagnesium chloride in THF followed by

p-toluenesulfonic acid-mediated dehydration and subsequent p-chloranil oxidative dehydrogenation gave 4-(4-pentyl-4-phenyl -4-silacyclohexyl)-4'-(trans-4-propylcyclohexyl)biphenyl (III). Chlorination of III with BrCl in CCl4 followed by LiAlH4 redn. in THF gave title compd., trans,trans-4-(4-pentyl-4-silacyclohexyl)-4'-(4-propylcyclohexyl)biphenyl. I are useful in liq. crystal displays.

L9 ANSWER 12 OF 70 CAPLUS COPYRIGHT 2003 ACS on STN

AN 1994:509812 CAPLUS

DN 121:109812

TI Synthesis, absorption characteristics and some reactions of polygermanes

AU Mochida, Kunio; Chiba, Hiromi

CS Department of Chemistry, Faculty of Science, Gakushuin University, 1-5-1 Mejiro, Toshima-ku, Tokyo, 171, Japan

SO Journal of Organometallic Chemistry (1994), 473(1-2), 45-54 CODEN: JORCAI; ISSN: 0022-328X

DT Journal

LA English

AB A no. of high mol. wt. polygermanes were prepd. by an improvement on Wurtz coupling reactions of dichlorogermanes and Na metal, and by a method using GeI2 and Grignard reagents (or organolithiums). Most of the polygermanes thus prepd. showed a narrow mol. distribution with mol. wts. 103-104. In soln., the polygermanes showed characteristic electronic absorption bands at 300-350 nm and were strongly thermochromic for alkyl-substituted derivs. Photolysis of the polygermanes proceeded by both contraction of the chain with loss of diorganogermylenes and homolytic scission of the Ge-Ge bond.

L9 ANSWER 13 OF 70 CAPLUS COPYRIGHT 2003 ACS on STN

AN 1994:107204 CAPLUS

DN 120:107204

Organomercury compounds. XXXI. Preparations and 199Hg NMR spectra of organomercury derivatives of 2-phenylpyridine, benzo[h]quinoline, 1-phenylpyrazole and 3,4,5-trimethyl-1-phenylpyrazole, and the x-ray crystal structure of bis[2-(pyridin-2'-yl)phenyl]mercury

AU Black, David St. C.; Deacon, Glen B.; Edwards, Gavin L.; Gatehouse, Bryan

CS Sch. Chem., Univ. New South Wales, Kensington, 2033, Australia

SO Australian Journal of Chemistry (1993), 46(9), 1323-36 CODEN: AJCHAS; ISSN: 0004-9425

DT Journal

LA English

OS CASREACT 120:107204

GΙ

2-(Pyridin-2'-yl)phenylmercuric acetate has been prepd. by mercuration of AΒ .2-phenylpyridine. Symmetrization of the corresponding chloride by alk. sodium stannite gave bis[2-(pyridin-2'-yl)phenyl] mercury (I), which was also prepd. from 2-(2'-aminophenyl)pyridine by the diazo method and treatment of the initial product with copper powder and aq. ammonia. Mercuration of benzo[h]quinoline and 3,4,5-trimethyl-1-phenylpyrazole with mercuric acetate followed by treatment with lithium chloride yielded benzo[h]quinolin-10-ylmercuric chloride and 2-(3',4',5'trimethylpyrazol-1'-yl)phenylmercuric chloride, resp. Treatment of the former product with tribromide ions gave 10-bromobenzo[h] quinoline. The exchange Grignard reaction between 1-phenylpyrazole and ethylmagnesium bromide to give 2-(pyrazol-1'-yl)phenylmagnesium bromide has been monitored by reactions with benzonitrile and D2O to establish optimum conditions for reaction with mercuric bromide giving bis[2-(pyrazol-1'-yl)phenyl]mercury. The 199Hg NMR chem. shifts of the majority of mercurials are shifted substantially downfield relative to the corresponding simple phenylmercurials consistent with weak intramol. coordination by the heterocyclic nitrogen donor atoms, but a small upfield shift is obsd. for bis[2-(pyrazol-1'-yl)phenyl]mercury. The x-ray crystal structure of I shows a centrosym. mol. with strong linear two coordination [Hg-C 2.098(8).ANG.; C-Hg-C 180.0.degree.] and significant but much weaker Hg-N interactions [Hg-N 2.798(7).ANG.; N-Hg-N 180.0.degree.] giving overall distorted square planar stereochem. The Ph rings are mutually coplanar, while the two pyridin-2'-yl rings are parallel and inclined at 10.8.degree. to the Ph groups.

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L9 ANSWER 14 OF 70 CAPLUS COPYRIGHT 2003 ACS on STN
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FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
ΡI	EP 501702	A2	19920902	EP 1992-301489	19920221
	EP 501702	A3	19931208		
	R: DE, FR,	GB, IT	1 •		
				GB 1991-4050	19910227
	CA 2061770	AA	19920828	CA 1992-2061770	19920225
				GB 1991-4050	19910227
	JP 05086082	A2	19930406	JP 1992-76166	19920227
				GB 1991-4050	19910227
	US 5298663	Α	19940329	US 1993-35524	19930323
				GB 1991-4050	19910227
			i	US 1992-840353	19920224
	US 5414133	Α	19950509	US 1994-179403	19940110
	•			GB 1991-4050	19910227
				US 1992-840353	19920224
				US 1993-35524	19930323

OS CASREACT 117:212725; MARPAT 117:212725

AN 1992:612725 CAPLUS

DN 117:212725

TI Process for the preparation of protected phosphine oxides from phosphinate esters and organomagnesium halides or organolithium compounds

IN Hall, Roger Graham; Riebli, Peter

PA Ciba-Geigy A.-G., Switz.

SO Eur. Pat. Appl., 11 pp. CODEN: EPXXDW

DT Patent

LA English

AB Protected phosphine oxides were prepd. by reaction of protected

phosphinate esters with organomagnesium halides or organolithiums at -70 -+65.degree. Thus, EtOP(0)HCH2(OEt)2 was added to MeMgBr in THF at 0-5.degree. to give MeP(0)HCH2(OEt)2.

- L9 ANSWER 15 OF 70 CAPLUS COPYRIGHT 2003 ACS on STN
- AN 1992:550944 CAPLUS
- DN 117:150944
- TI Additions of 1-(.alpha.-aminoalkyl)benzotriazoles to enol ethers. New routes to 1,3-amino ethers
- AU Katritzky, Alan R.; Rachwal, Stanislaw; Rachwal, Bogumila; Steel, Peter J.
- CS Dep. Chem., Univ. Florida, Gainesville, FL, 32611-2046, USA
- SO Journal of Organic Chemistry (1992), 57(18), 4932-9 CODEN: JOCEAH; ISSN: 0022-3263
- DT Journal
- LA English
- OS CASREACT 117:150944

GΙ

- AB 1-(.alpha.-Aminoalkyl)benzotriazoles add readily to enol ethers to give the corresponding 1-benzotriazolyl-3-aminoalkyl ethers, e.g. I (R1 = Ph, Me2CH) in high yields. Subsequent replacement of the benzotriazole moiety by an alkyl or aryl group (with a Grignard reagent) or by a hydrogen atom (with lithium aluminum hydride) affords 1,3-amino ethers in good yields: Anchimeric assistance by the amino groups in the substitutions of the benzotriazolyl moiety facilitates the reactions. Full stereochem. is assigned to the stereoisomeric products on the basis of NMR techniques and x-ray diffraction.
- L9 ANSWER 16 OF 70 CAPLUS COPYRIGHT 2003 ACS on STN
- AN 1991:582681 CAPLUS
- DN 115:182681
- TI Intramolecular palladium-catalyzed trimethylenemethane cycloadditions: initial studies
- AU Trost, Barry M.; Grese, Timothy A.; Chan, Dominic M. T.
- CS Dep. Chem., Stanford Univ., Stanford, CA, 94305, USA
- SO Journal of the American Chemical Society (1991), 113(19), 7350-62 CODEN: JACSAT; ISSN: 0002-7863
- DT Journal
- LA English
- OS CASREACT 115:182681

GI

AB The potential application of [3 + 2] cycloaddns. to polycarbocycle construction is considerably enhanced by the ability to perform such reactions intramolecularly. The feasibility of such processes is explored in the context of Pd-catalyzed cycloaddns. of 2-[(trimethylsilyl)methyl]allyl carboxylates, e.g., Me3SiCH2C(:CH2)CH(OAc)CH2CH2CH2CH:CHCO2Et, wherein the trimethylenemethane (TMM) precursor fragment (donor) and the electron-deficient olefin (acceptor) are joined by a tether of simple methylene groups of 3, 4, 5, and 8 members. Several versatile synthetic routes to these substrates were developed. 2-Bromo-3-(trimethylsilyl)propene proves to be a key reagent for construction of the donor portion. Acceptors bearing esters, cyano groups, and esp. sulfones were examd. The diastereoselectivity of the reaction was explored both in terms of ring juncture and the diastereofacial selectivity with respect to an oxygen substituent at the allylic position of the acceptor. Excellent cycloaddns, to give the bicyclo[3.3.0]octyl and bicyclo[4.3.0]nonyl systems, e.g., I (R = CO2Et, SO2Ph) and II, are obsd., whereas larger rings cannot be obtained in this series. The choice of catalyst proves crit., the most useful being either Pd(PPh3)4 and DPPE or, more generally, (Me2CHO)3P and Pd(OAc)2. The first cycloaddn. of a 1,1-dialkylated TMM precursor, which fails in intermol. cases, was obsd. in this intramol. series to give a bridgehead-substituted bicycle. A rationale for the obsd. diastereoselectivity is presented.

- L9 ANSWER 17 OF 70 CAPLUS COPYRIGHT 2003 ACS on STN
- AN 1991:492231 CAPLUS
- DN 115:92231
- Organometallic reactions characteristic of chiral heterocyclic compounds: synthesis and stereoselective **Grignard reaction** of chiral 4-oxa-7,7a-diazaperhydroindans
- AU Takahashi, Hiroshi; Senda, Takashi; Higashiyama, Kimio
- CS Fac. Pharm. Sci., Hoshi Univ., Tokyo, 142, Japan
- SO Chemical & Pharmaceutical Bulletin (1991), 39(4), 836-42 CODEN: CPBTAL; ISSN: 0009-2363
- DT Journal
- LA English
- OS CASREACT 115:92231

GΙ

AB New heterocyclic compds., 6-phenyl-4-oxa-7,7a-

diazaperhydroindans I (R = Me, CHMe2), were synthesized by condensation of chiral (2-hydroxyethyl)hydrazines II, prepd. from (R)-phenylglycinol, with .gamma.-chlorobutyraldehyde. The stereoselective ${\bf Grignard}$ ${\bf reaction}$ of I afforded chiral 2-substituted 1-[N-(2-hydroxy-1-phenylethyl)amino]pyrrolidines (III; R1 = Me, Et, Ph, CH2Ph). The mol. structure of I (R = Me) was detd. by x-ray crystallog. anal.

- L9 ANSWER 18 OF 70 CAPLUS COPYRIGHT 2003 ACS on STN
- AN 1991:247788 CAPLUS
- DN 114:247788
- TI Peptide derivatives preparation as retroviral protease inhibitors
- IN Kempf, Dale J.; Plattner, Jacob J.; Norbeck, Daniel W.; Boyd, Steven A.; Baker, William R.; Erickson, John W.; Fung, Anthony K. L.; Crowley, Steven R
- PA Abbott Laboratories, USA
- SO PCT Int. Appl., 222 pp. CODEN: PIXXD2
- DT Patent
- LA English

FAN.CNT 1

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										W	O 198	89-US	2055	19890	512
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- OS MARPAT 114:247788
- AB Peptide derivs. are prepd. as retroviral protease inhibitors. Synthetic processess involved carbodiimide coupling, or coupling in combination with deprotection, and reaction with mixed anhydrides. Thus, N-methyl-1-cyclohexenecarboxamide was treated with BuLi in THF, treated with ClTi(OPr-iso)3, and then Boc-phenylalaninal to give N-methyl-6-[2-(tert-butoxycarbonyl)amino-1-hydroxy-3-phenyl]propyl-1-cyclohexenecarboxamide. This was then deprotected with HCl in dioxane to give N-methyl-6-(2-amino-1-hydroxy-3-phenylpropyl)-1-cyclohexenecarboxamide-HCl (I). I was coupled with Boc-Leu-Asn in the presence of 180-BuO2CCl to give the amide.
- L9 ANSWER 19 OF 70 CAPLUS COPYRIGHT 2003 ACS on STN
- AN 1990:440676 CAPLUS
- DN 113:40676
- TI 1-[(2-fluorophenyl)(4-fluorophenyl)phenylmethyl]-1H-imidazole and related medical fungicides
- IN Bartroli, Javier; Anguita, Manuel

PA Uriach, J., y Cia. S. A., Spain

SO Eur. Pat. Appl., 19 pp.

CODEN: EPXXDW

DT Patent

10085368.23

LA English

FAN.CNT 1

IAW.	PATEN	T NO.		KIND	DATE		API	PLICATION NO.	DATE
PI	EP 35			A1 B1	1990013 1991101	_	EP	1988-112239	19880728
	R	: AT,	BE,	CH, DE	, FR, GB	, GR,	,	LI, LU, NL, SE	
	AT 68	486		E	1991111	5		1988-112239 1988-112239	19880728 19880728
		048569		A2	1990021	-	JP	1988-268344	19881026
	JP 06	025145		B4	1994040	6	EP	1988-112239	19880728
	CA 13	27589		A1	1994030	8	CA	1988-582916	19881114
	115 51	49707		А	1992092	2		1988-112239 1990-628937	19880728 19901214
	05 51	43707		A	1772072			1988-112239	19880728
							US	1988-257095	19881013

OS CASREACT 113:40676; MARPAT 113:40676

GI

The title compd. (I) (UR-4506) (and related compds.) were prepd. by reaction of [(2-fluorophenyl)(4-fluorophenyl)phenyl] chloromethane (II) (or the correspong trityl chlorides) with imidazole or Li imidazolide in a polar, inert solvent (MeCN) at 0.degree.-reflux for 5 min-3 h. Thus, PhMgBr reacted with 2,4'-difluorobenzophenone to give 97% of the tritylcarbinol, which was converted to II (83%) with SOCl2. II was stirred with Li imidazolide in MeCN at room temp. for 2 h to give 96% I. I had an MIC of 1.0 .mu.g/mL against Candida albicans ATCC 10231. I formulations are given.

- L9 ANSWER 20 OF 70 CAPLUS COPYRIGHT 2003 ACS on STN
- AN 1990:7568 CAPLUS
- DN 112:7568
- TI Synthetic route to poly(sily1) methanes via poly(phenylsily1) methanes and poly(bromosily1) methanes
- AU Hager, Rudolf; Steigelmann, Oliver; Mueller, Gerhard; Schmidbaur, Hubert
- CS Anorg.-Chem. Inst., Tech. Univ. Muenchen, Garching, D-8046, Fed. Rep. Ger.

- SO Chemische Berichte (1989), 122(11), 2115-19 CODEN: CHBEAM; ISSN: 0009-2940
- DT Journal
- LA English
- OS CASREACT 112:7568
- A three-step synthesis is presented for di- and tri(silyl)methane, two AΒ feedstock gases for the chem. vapor deposition of amorphous hydrogenated silicon/carbon alloys (a-SiC:H). Chloro(phenyl)silane and dior trihalomethanes react with magnesium in THF to give high yields of bis- and tris(phenylsilyl) methane, resp. The two products can be converted into bis- and tris(bromosilyl) methane by treatment with anhyd. hydrogen bromide. Bromide/hydride substitution in these precursors is accomplished with lithium aluminum hydride in a two-phase system using a phase-transfer catalyst. The compds. CH2(SiH2Ph)2, CH(SiH2Ph)3, CH2(SiH2Br)2, CH(SiH2Br)3, CH2(SiH3)2, and CH(SiH3)3 have been characterized by std. spectroscopic methods, and the crystal and mol. structure of CH(SiH2Ph)3 has been detd. by single-crystal x-ray diffraction. The mol. adopts a conformation with crystallog. C3 symmetry. This result is discussed with regard to the structure of related mols. with three substituents of potential Cs symmetry at a tetrahedral center.
- L9 ANSWER 21 OF 70 CAPLUS COPYRIGHT 2003 ACS on STN
- AN 1989:614170 CAPLUS
- DN 111:214170
- TI Derivatives of tamoxifen. Dependence of antiestrogenicity on the 4-substituent
- AU McCague, Raymond; Leclercq, Guy; Legros, Nicole; Goodman, Joyce; Blackburn, G. Michael; Jarman, Michael; Foster, Allan B.
- CS Drug Dev. Sect., Inst. Cancer Res., Sutton/Surrey, SM2 5PX, UK
- SO Journal of Medicinal Chemistry (1989), 32(12), 2527-33 CODEN: JMCMAR; ISSN: 0022-2623

Ι

- DT Journal
- LA English
- OS CASREACT 111:214170

GΙ

AB A range of tamoxifen (I; R = H) derivs. substituted in the 4-position of the 1-Ph ring are described. The key steps in the synthesis of I (R = iodo, Br, MeS) were reactions of 1,2-diarylbutanones with the (4-halophenyl)lithium or [4-(methylthio)phenyl]

magnesium bromide. Oxidized precursors of 4-(methylthio)tamoxifen were used to prep. the methylsulfinyl and methylsulfonyl derivs. Further derivs. I (R = formyl, hydroxymethyl, oxiranyl, mercapto) were prepd. from 4-bromotamoxifen via the 4-lithio deriv. Several of the derivs. I (R =

Br, iodo, SMe, SOMe, SO2Me, oxiranyl, CHO, CH2OH) displayed a higher affinity for estrogen receptors (ER) of calf uterine cytosol than did tamoxifen, but there was no relationship between affinity to ER and the ability to inhibit the growth of the MCF-7 breast cancer cell line in vitro.

- L9 ANSWER 22 OF 70 CAPLUS COPYRIGHT 2003 ACS on STN
- AN 1988:493295 CAPLUS
- DN 109:93295
- TI Structural characterization in solution of intermediates in rhodium-catalyzed hydroformylation and their interconversion pathways
- AU Brown, John M.; Kent, Alexander G.
- CS Dyson Perrins Lab., Oxford, OX1 3QY, UK
- SO Journal of the Chemical Society, Perkin Transactions 2: Physical Organic Chemistry (1972-1999) (1987), (11), 1597-607 CODEN: JCPKBH; ISSN: 0300-9580
- DT Journal
- LA English
- OS CASREACT 109:93295

GI

- AB The reaction of HRh(CO)(PPh3)3 (I) with CO has been studied by 1H, 13C, and 31P NMR. The main species present under ambient conditions is HRh(CO)2(PPh3)2 (II) which exists as two rapidly equilibrating trigonal bipyramidal isomers. Complexes I and II are in rapid equil. via CO and PPh3 dissocn. steps and the square-planar complexes HRh(CO)(PPh3)2 (III) and HRh(CO)2PPh3 (IV) are likely transient intermediates. The chem. of these PPh3 complexes is compared with that of closely related 5phenyl-5H-dibenzophosphole and 1,3-bis(diphenylphosphino)propane analogs. Complex I catalyzes the isomerization of (Z)-[1,2-2H2] styrene, effectively suppressed by CO or PPh3. HRh(CO)2P2 complexes trap methylenecyclopropane. In the presence of styrene and CO, I is converted into a branched acyl deriv., e.g. V, which readily equilibrates with its linear isomer VI; the stereochem. of these acyl derivs. is detd. by low-temp. NMR; at higher temps. rapid inter- and intramol. exchange processes occur. The relevance of these observations to Rh-catalyzed hydroformylation is discussed and it is proposed that the regiochem. of reaction is largely controlled by competitive olefin trapping involving complexes III and IV.
- L9 ANSWER 23 OF 70 CAPLUS COPYRIGHT 2003 ACS on STN
- AN 1984:139172 CAPLUS
- DN 100:139172
- TI Stereoselectivity in the condensation reactions of 1-phenylethyl alkyl and phenyl ketones with organometallic reagents
- AU Alvarez-Ibarra, Carlos; Arjona, Odon; Perez-Ossorio, Rafael; Perez-Rubalcaba, Alfredo; Quiroga, Maria L.; Santesmases, Maria J.
- CS Dep. Quim. Org., Univ. Complutense, Madrid, Spain
- SO Journal of the Chemical Society, Perkin Transactions 2: Physical Organic Chemistry (1972-1999) (1983), (11), 1645-8

10085368.23 Page 128

CODEN: JCPKBH; ISSN: 0300-9580

- DT Journal
- LA English
- OS CASREACT 100:139172
- AB The stereoselectivity of the condensation reactions of PhCHMeCOR (R = Me, Et, CHMe2, CMe3, Ph) with organomagnesium and organolithium derivs. in ethers was examd. Results are accounted for on the basis of competition between 2 transition states which may adopt either Karabatsos- or Felkin-type conformations according to the nature of R, the reagent nucleophilicity, and the polarity of the solvent. Polar and steric anal. of this reaction allows highly stereoselective syntheses of diastereoisomeric .alpha.-phenylalkanols to be devised.
- L9 ANSWER 24 OF 70 CAPLUS COPYRIGHT 2003 ACS on STN
- AN 1982:563050 CAPLUS
- DN 97:163050
- TI The stereochemistry of condensation reactions of (.+-.)-3-phenylbutan-2one with phenylmetallic compounds as a function of the reagent nucleophilicity
- AU Arjona, Odon; Perez-Ossorio, Rafael; Perez-Rubalcaba, Alfredo; Quiroga, Maria L.
- CS Dep. Quim. Org., Univ. Complutense, Madrid, 3, Spain
- SO Journal of the Chemical Society, Chemical Communications (1982), (8), 452-3
 - CODEN: JCCCAT; ISSN: 0022-4936
- DT Journal
- LA English
- OS CASREACT 97:163050
- AB The stereoselectivity of nucleophilic addn. reactions of (.+-.)-MeCHPhCOMe with PhMgBr, Ph2Mg, PhLi, and Ph3Al was studied. The percentage of (R,S)-MeCHPhCPh(OH)Me (I) increased in the order Al < Mg < Li. Addn. of LiClO4 to the reaction mixt. caused small increases in stereoselectivity in favor of I; similar effects were obsd. on increasing the polarity of the solvent. In the presence of CuI or FeCl3 the percentage of I decreased, indicating a partially radical mechanism.
- L9 ANSWER 25 OF 70 CAPLUS COPYRIGHT 2003 ACS on STN
- AN 1982:143137 CAPLUS
- DN 96:143137
- TI New approach to Lythraceae alkaloids: total synthesis of (.+-.)-vertaline
- AU Hart, David J.; Kanai, Kenichi
- CS Dep. Chem., Ohio State Univ., Columbus, OH, 43210, USA
- SO Journal of Organic Chemistry (1982), 47(8), 1555-60 CODEN: JOCEAH; ISSN: 0022-3263
- DT Journal
- LA English

GΙ

- AB A new approach to Lythraceae alkaloids is described within the context of a total synthesis of (.+-.)-vertaline (I). The use of N-silyl imines in the prepn. of benzylic amines as well as a stereoselective bicycloannulation approach to the synthesis of quinolizidinones is discussed.
- L9 ANSWER 26 OF 70 CAPLUS COPYRIGHT 2003 ACS on STN
- AN 1981:497239 CAPLUS
- DN 95:97239
- TI Vinyl selenides and selenoxides: preparation, conversion to **lithium** reagents, Diels-Alder reactivity, and some comparisons with sulfur analogs
- AU Reich, Hans J.; Willis, Willam W., Jr.; Clark, Peter D.
- CS Dep. Chem., Univ. Wisconsin, Madison, WI, 53706, USA
- SO Journal of Organic Chemistry (1981), 46(13), 2775-84 CODEN: JOCEAH; ISSN: 0022-3263
- DT Journal
- LA English
- A variety of aryl vinyl selenides are prepd by reaction of vinyl Grignard AB reagents with aryl selenenyl bromides, or by reductive elimination of the adducts of [bis(arylseleno)methyl]lithiums with carbonyl compds. Ph vinyl selenide is deprotonated with LiN(CHMe2)2 at -78.degree. in THF. Vinyl selenides with .beta.-alkyl groups require Li tetramethylpiperidide (I) and higher temps. (-50.degree.) for complete deprotonation. Allylic Li reagents were obtained from 1-propenyl and 2-methyl-1-propenyl selenides whereas 1-butenyl or 3-methyl-1-butenyl selenides gave vinyllithium reagents. Reaction with electrophiles proceeds in good to excellent yield. Primary halides require (Me2N) 3PO to react well. Unhindered carbonyl compds. react without enolization. Deprotonation with LiN(CHMe2)2 is reversible, and during competitive deprotonation studies with LiN(CHMe2)2 aryl vinyl sulfides are thermodn. less acidic than aryl vinyl selenides. Deprotonation with I is irreversible, and competitive deprotonation studies showed vinyl selenide to be kinetically more acidic as well. m-F3CC6H4SCH2CH:CH2, as expected, is more acidic than the Se compd. Vinyl selenoxides can be prepd with 3-ClC6H4CO2OH. They are not thermally stable enough to serve as acetylene equiv. in Diels-Alder reactions. PhSeCH: CH2 gives a Diels-Alder addn. product with 1,4-diphenylisobenzofuran but failed to give cycloaddn. products with less reactive dienes. PhSeOCH: CH2 does not give a useful yield of Li reagent upon reaction with amide bases.
- L9 ANSWER 27 OF 70 CAPLUS COPYRIGHT 2003 ACS on STN

AN 1980:567250 CAPLUS

DN 93:167250

TI The photochemistry of 4-phenyl-1-iodobutane and 4-phenyl -2-iodomethyl-1-butene

- AU Charlton, James Leslie; Williams, Gaynor Jane; Lypka, Gerald Nicholas
- CS Dep. Chem., Univ. Manitoba, Winnipeg, MB, R3T 2N2, Can.
- SO Canadian Journal of Chemistry (1980), 58(12), 1271-4 CODEN: CJCHAG; ISSN: 0008-4042

DT Journal

LA English

- AB The photochem. of 4-phenyl-1-iodobutane (I) and 4-phenyl
 -2-iodomethyl-1-butene (II) is examd. to det. the effect of structure on
 the character of the intermediates generated on photolysis. The irradn.
 of I gives only radical intermediates. By contrast the photolysis of II
 gives products that are more consistent with cationic intermediates.
- L9 ANSWER 28 OF 70 CAPLUS COPYRIGHT 2003 ACS on STN

AN 1979:483017 CAPLUS

DN 91:83017

- TI Synthesis and biological activity of cocaine analogs. 2. 6H-[2]Benzopyrano[4,3-c]pyridin-6-ones
- AU Lazer, Edward S.; Hite, Gilbert J.; Nieforth, Karl A.; Stratford, Eugene S.
- CS Sch. Pharm., Univ. Connecticut, Storrs, CT, 06268, USA
- SO Journal of Medicinal Chemistry (1979), 22(7), 845-9 CODEN: JMCMAR; ISSN: 0022-2623
- DT Journal
- LA English

GI

- AB 1,2,3,4-Tetrahydro-2-methyl-6H-[2]benzopyrano[4,3-c]pyridin-6-one (I) [70932-75-1], cis- (II) [70932-81-9], and trans- 1,3,4,4,4a.alpha.,10b.beta.-hexahydro-2-methyl-6H-[2]benzopyrano[4,3-c]pyridin-6-one (III) [70932-77-3] were prepd. and evaluated for biogenic amine uptake inhibition. In comparison to cocaine and tropacocaine the 3 prepd. compds showed weak inhibition of norepinephrine [51-41-2] uptake by rat brain synaptosomal prepns. III showed 36% inhibition, whereas II was ineffective.
- L9 ANSWER 29 OF 70 CAPLUS COPYRIGHT 2003 ACS on STN

AN 1976:105530 CAPLUS

DN 84:105530

TI Heterocyclic analogs of pleiadine. XIX. Reaction of perimidine

derivatives with organometallic compounds

- AU Pozharskii, A. F.; Smirnova, L. P.; Tertov, B. A.; Kashparov, I. S.; Sokolov, V. I.
- CS Rostov. Gos. Univ., Rostov-on-Don, USSR
- SO Khimiya Geterotsiklicheskikh Soedinenii (1975), (12), 1682-7 CODEN: KGSSAQ; ISSN: 0132-6244
- DT Journal
- LA Russian

GI

- Dihydroperimidines (I, R = Bu, Ph, 2-thienyl, Rl = H) were obtained in 84-98% yields by treatment of perimidine (II, R = H) with BuLi, PhLi, 2-thienyllithium, and PhMgBr. Also obtained was 5% 1-methylperimidine-2-carboxylic acid. Analogously obtained were 60 and 77% I (R = Rl = Bu, Ph). Treatment of II (R = Cl) with BuLi and PhLi gave 28-47% II (R = Bu, Ph). Addnl. obtained was 90-1% III (R = Me, Ph, 2-thienyl).
- L9 ANSWER 30 OF 70 CAPLUS COPYRIGHT 2003 ACS on STN
- AN 1975:459799 CAPLUS
- DN 83:59799
- TI Aromatic hydrocarbon polymers
- IN Hay, Allan S.; Relles, Howard M.
- PA General Electric Co.
- SO U.S., 3 pp. CODEN: USXXAM
- DT Patent
- LA English
- FAN. CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	US 3810879	Α	19740514	US 1972-240786	19720403
				US 1972-240786	19720403

- AB Arom. diolefins, such as 2,2-bis(4-phenyl-3-cyclohexen-1-yl)propane (I), were manufd. by Grignard reaction, and polymd. in the presence of Li to give arom. hydrocarbon polymers, such as poly[2,2-bis(4-phenyl-3-cyclohexen-1-yl)propane] (II) useful for dielectrics. Thus, a mixt. of I 10 (by reaction of 2,2-bis(4-oxocyclohexyl)propane with bromophenyl Mg) and Li 6 in THF 200 parts was stirred for 11 hr at .apprx.25.degree. in N, and treated with 510 parts CH3OH to ppt. II with 90% yield and 26,500 mol. wt.
- L9 ANSWER 31 OF 70 CAPLUS COPYRIGHT 2003 ACS on STN
- AN 1974:551268 CAPLUS
- DN 81:151268
- TI Quasi-Favorskii rearrangement. Synthesis of 1-phenylcycloalkanecarboxylic

- AU Stevans, Calvin L.; Pillai, P. Madhavan; Taylor, K. Grant
- CS Dep. Chem., Wayne State Univ., Detroit, MI, USA
- SO Journal of Organic Chemistry (1974), 39(21), 3158-61 CODEN: JOCEAH; ISSN: 0022-3263
- DT Journal
- LA English
- .alpha.-Halo ketones without an .alpha.-H undergo C skeleton AΒ rearrangements on treatment with the Li salt of arom. primary amines in ether, yielding amides. The action of Li anilide on 1-benzoyl-1bromocyclohexane gave 55% 1-phenyl-cyclohexanecarboxanilide and 30% 1-benzoyl-1-(phenylamino)-cyclohexane. The yield of the rearranged amide was improved by using a more hindered amine such as .omicron.-toluidine or 2,6-dimethylaniline. Also, a p-MeO substituent on the phenyl group of the .alpha.-halo ketone facilitated the rearrangement while a m-Cl substituent decreased the yield of the rearranged amide. The extension of this rearrangement to .alpha.-bromocycloalkyl Ph ketones and hydrolysis of the resulting amides gave 1-phenylcycloalkane-carboxylic acids. Treatment of endo-2-benzoyl-exo-2-bromonorborane with Li anilide gave endo-2-phenylnorbornane-exo-2-carboxanilide, as expected from a concerted semibenzilic rearrangement mechanism.
- L9 ANSWER 32 OF 70 CAPLUS COPYRIGHT 2003 ACS on STN
- AN 1974:15004 CAPLUS
- DN 80:15004
- TI Reaction of lithium phenyl- and vinylacetylenides with trimethylchloro- and trimethylfluorosilanes
- AU Florensova, O. N.; Volkova. L. I.; Maroshin, Yu. V.; Kryazhev, Yu. G.
- CS Irkutsk. Inst. Org. Khim., Irkutsk, USSR
- SO Zhurnal Obshchei Khimii (1973), 43(9), 1992-3 CODEN: ZOKHA4; ISSN: 0044-460X
- DT Journal
- LA Russian
- AB Treating Mg and Li derivs. of PhC.tplbond.CH and CH2:CHC.tplbond.CH with Me3SiCl and Me3SiF to give RC.tplbond.CSiMe3 (R = Ph, CH2:CH). The best yields were obtained from Li acetylides and Me3SiF (71-4%).
- L9 ANSWER 33 OF 70 CAPLUS COPYRIGHT 2003 ACS on STN
- AN 1968:506783 CAPLUS
- DN 69:106783
- TI Reaction of organolithium and organomagnesium compounds with carbonyl derivatives of barenes and neobarenes
- AU L'vov, A. I.; Zakharkin, L. I.
- CS Inst. Elementoorg. Soedin., Moscow, USSR
- SO Izvestiya Akademii Nauk SSSR, Seriya Khimicheskaya (1967), (12), 2653-62 CODEN: IASKA6; ISSN: 0002-3353
- DT Journal
- LA Russian
- GI For diagram(s), see printed CA Issue.
- AB Tertiary carboranyl alcs. are cleaved at C-C bond by catalytic amts. of RONa. Reaction of MeMgI with 0.3 mole 1-methyl-2-acetylcarborane (I) in Et2O gave 53% starting ketone, 42% methylcarboranyl(dimethyl)carbinol (II) and 4.8% methylcarborane (III); MeLi similarly gave 84% starting ketone and 16% III; similar reaction in tetrahydrofuran (THF) gave from MeMgI 43% starting ketone, 32% III and 25% II, while with MeLi 90% III was formed and 10% unreacted ketone was recovered. Methylcarboranylmagnesium bromide (IIIa) in THF treated with Me2CO 3 hrs. gave 8% II and 92% III. II kept overnight with EtONa in EtOH gave no residual II and a considerable amt.

of III. MeMgI and methylcarboranylcarboxylyl chloride in Et2O gave 30% II, m. 87-9.degree.. EtMgBr and I in Et2O gave in 0.5 hr. 94% 1-methylcarboranylethanol, m. 153-4.degree.. 1-Methyl-7acetylneocarborane and MeMgI in Et20 0.5 hr. gave 91.5% starting ketone and 8.5% methylneocarboranyldimethylcarbinol (IV); MeLi similarly gave 55% above carbinol and 3% methylneocarborane, besides 42% initial ketone. Methylneocarboranyllithium (V) in C6H6 gave with Me2CO (20% excess) in 2 hrs. 95% IV, m. 29-30.degree.. MeMgI and 1-methyl-7-benzoylneocarborane gave in 2 hrs. in Et2O 62% (methylneocarboranyl)phenylmethylcarbinol, bl.cntdot.5 152-3.degree., n20D 1.5807, also obtained from AcPh and V. Similar reaction with PhMgBr gave 88% (methylneocarboranyl)diphenylcarbino 1, m. 90-1.degree.. MeMgI and 2,3-carborano-4,5-benzocyclopentanone in Et2O gave 82% VI m. 140-1.degree.; MeLi gave 76% VI, while PhLi gave 77% VII, m. 165-6.degree.. Bis(phenylcarboranyl) ketone and MeMgI in Et20 gave 82% bis(phenylcarboranyl)carbinol, m. 272-4.degree., while PhMgBr gave in 10 hrs. at 45.degree. in Et20-C6H6 66.7% same carbinol. Phenyl-2-benzoylcarborane (VIII) and MeMqI gave 45% (phenylcarboranylmethyl) methylcarbinol, m. 128-9.degree., while the filtrate from this was treated with CrO3 to yield VIII, phenyl (phenylcarboranyl)carbinol (IX) and (phenylcarboranyl)phenylmethylcarbinol , all isolated as acetates after acetylation with Ac20. PhMgBr and VIII in THF gave 88% IX m. 122-4.degree., and some phenylcarborane (X); similar reaction in Et20 gave Ph2, X and Ph3COH as well as [o-PhCB10H10CC(OH)Ph]2, m. 239-40.degree.. PhLi and methylcarboranecarboxaldehyde (XI) in Et20 0.5 hr. gave 85% (methylcarboranyl)phenylcarbinol (XII), m. 107.degree.; the same formed from this reaction in THF. IIIa and XIa in Et2O gave 93.5% 1-(methylneocarboranyl)-2-carboranyl-1-ethanol, m. 239-40.degree... The appropriate aldehyde and methylcarboranyllithium gave 95% bis(methylcarboranyl)carbinol, m. 177-8.degree.. XII and EtONa-EtOH in 12 hrs. at 20.degree. gave III as the sole product. iso-PrMgBr and XI in Et20 gave 92% methylcarboranylcarbinol, m. 268-9.degree. Ozonolysis of vinyl or allyl carborane or neocarborane and treatment of the mixt. with Me2S at -30.degree. gave after warming and an aq. treatment the following aldehydes RCHO: o-HCB10H10C, m. 212-13.degree. (2,4dinitrophenylhydrazone, m. 183-4.degree.); o-MeCB10H10C, m. 220-2.degree. (189.degree.); o-HCB10H10CCH2, m. 94-5.degree. (m. 163-4.degree.); o-MeCB10H10CCH2, m. 140-2.degree. (m. 176-7.degree.); m-MeCB10H10C, m. 143-4.degree. (m. 177-8.degree.); m-MeCB10H10CCH2, b2 98.degree., (m. 154-5.degree.). Carboranyllithium or corresponding RMqX in THF treated with ethoxymethyleneaniline and heated 1 hr. gave after an aq. treatment and steam distn. of the org. layer with dil. H2SO4 40-55% carboranecarboxaldehyde, XI and 25% XIa. (Methylneocarboranyl)methylcarbi nol acetate, b7 127-30.degree., gave 1-methyl-7-vinylneocarborane, m. 75-6.degree.. Allyl bromide added to V in C6H6 and heated 2 hrs. gave 80% 1-methyl-7-allylneocarborane, b7 95-6.degree., n22D 1.5260, d20 0.9040. Similarly was prepd. 80% 1-methyl-2-allylcarborane, b13 142-3.degree., d20 0.9295.

- L9 ANSWER 34 OF 70 CAPLUS COPYRIGHT 2003 ACS on STN
- AN 1968:496600 CAPLUS
- DN 69:96600
- TI Reactions of azides and fused-ring tetrazoles with organomagnesium halides and organolithium compounds
- AU Skripnik, L. I.; Pochinok, V. Ya.
- CS Kiev. Gos. Univ. im. Shevchenko, Kiev, USSR
- SO Khimiya Geterotsiklicheskikh Soedinenii (1968), (3), 474-7 CODEN: KGSSAQ; ISSN: 0132-6244

DT Journal

- LA Russian
- GI For diagram(s), see printed CA Issue.
- A soln. of 2 g. 6-phenylthiazolo[2,3-e]tetrazole (I) in 200 ml. Et20 was AΒ dropwise treated during 30 min. with equimol. amt. PhMgBr in 20 ml. Et2O, the mixt. heated 30 min., and worked up as usual and extd. with Et2O to give 1.82 g. phenyl(4-phenyl-2-thiazolyl)triazene, m. 159-60.degree. (EtOH). The latter was also obtained using PhLi in the reaction. Similarly 2 g. 4-chloro-2-azidobenzothiazole (II) treated with an equilmol. amt. PhMgBr afforded 2.6 g. phenyl (4-chloro-2-benzothiazolyl)triazene, m. 174.degree. (EtOH). The latter was also prepd. from 2.1 g. II and PhLi at -10.degree. Analogously, 1 g. I treated with 1.6 g. BuMgBr in Et2O during 10 min., the mixt. stirred for 10 min. and worked up gave 0.72 g. butyl (4-phenyl -2-thiazolyl)-triazene, m. 88.degree. (decompn.) (petroleum ether). The latter was also obtained using PhLi in the reaction. Analogously, 2.1 g. II and BuMgBr reacted to give 95% yield of 4-chloro-2-aminobenzothiazole, m. 208.degree., which was also obtained when MeMqI, PhCH2MqCl, or BuLi were used in the reaction. A soln. of 2 g. NaCN and 0.2 g. NaOH in 40 ml. 75% EtOH was treated with 1.9 g. 4-methyltetrazolo[1,5-b]benzothiazole, heated for 30 min., poured into 100 ml. water, and acidified with 50% AcOH to give 1.63 g. cyano(4-methyl-2-benzothiazolyl)triazene, m. 157.degree. (decompn.) (EtOH). Fused-ring tetrazoles are tautomers and the course of the reaction depends on azido-tetrazole equil., which is affected by the nature of the substituent in the N-contq. ring and by the chem. nature of the coreactant.
- L9 ANSWER 35 OF 70 CAPLUS COPYRIGHT 2003 ACS on STN
- AN 1967:443166 CAPLUS
- DN 67:43166
- TI Reaction of organometallic compounds with .alpha.-ethylenic acetals
- AU Quelet, Raymond; Broquet, Colette; D'Angelo, Jean
- CS Fac. Sci., Sorbonne, Paris, Fr.
- SO Comptes Rendus des Seances de l'Academie des Sciences, Serie C: Sciences Chimiques (1967), 264(15), 1316-19
 CODEN: CHDCAQ, ISSN: 0567-6541
- DT Journal
- LA French
- AB The mechanism of the action of organomagnesium compds. on .alpha.-ethylenic acetals was further studied by varying the relative proportions of acetal and RMgX in the condensation of BuMgBr on the di-Et acetal (I) of acrolein under conditions previously described (CA 66: 37182z). Similar expts. with mole ratios 1:2 acetal-RLi refluxed 2 hrs. generally gave better yields than with the corresponding organomagnesium compds. I treated with BuLi gave 1-ethoxy-1-heptene, b. 170.degree.; I with PhLi gave 1-ethoxy-3-phenyl-1-propene, b16 110.degree.; I with C8H17Li, 1-ethoxy-1-undecene, b32 142-5.degree., n18D 1.440 [2,4-dinitrophenylhydrazone (DNPH) corresponding to the undecanal m. 108.degree.]. Two .beta.-substituted .alpha.-ethylenic acetals were treated with BuLi: (1) the di-Et acetal of crotonaldehyde gave total transposition to 62% (19% cis and 81% trans) 1-ethoxy-3-methyl-1-heptene, b22 80-3.degree., n17D 1.4315 (3-methylheptanal deriv. b163-40, n18D 1.4210; 2,4-DNPH deriv. m. 73.degree.); and (2) the di-Et acetal of cinnamaldehyde gave a radical-type addn. to form 46% di-Et acetal of 2-benzylhexanal, b0.3 103-4.degree., n17D 1.4800 (2,4-DNPH deriv. m. 125.degree.).
- L9 ANSWER 36 OF 70 CAPLUS COPYRIGHT 2003 ACS on STN
- AN 1965:439004 CAPLUS

DN 63:39004 OREF 63:6968c-f

TI Dihydropyridines. IX. Reactions of various magnesium and lithium organic compounds with 3,5-dicyano-4-methylpyridine

AU Kuthan, J.; Bartonickova, R.

CS Vysoka Skola Chem. Technol., Prague

SO Collection of Czechoslovak Chemical Communications (1965), 30(8), 2609-14 CODEN: CCCCAK; ISSN: 0010-0765

DT Journal

LA German

GI For diagram(s), see printed CA Issue.

cf. CA 61, 9461a, 10655a. MeMgBr, BuMgBr, PhMgBr, and PhCH2MgBr (from AΒ 0.03 mole Mg and 0.03 mole halogen deriv.) allowed to react as usual with 0.008 mole title compd. (I) gave compds. of type II (R, yield, m.p., solvent, .lambda.(EtOH), and .nu.(CHCl3) given): Me, 87%, 119-20.degree., C6H6, -, -; Bu, 90%, 86-7.degree., C6H6-cyclohexane, 217 m.mu. (log .epsilon. 4.28), 258 m.mu. (log .epsilon. 3.90), 372 m.mu. (log .epsilon. 3.60), 1556, 1631 cm.-1 (C:C), 2202 cm.-1 (C:N), 3429 cm.-1 (free NH), 3291 cm.-1 (bound NH); Ph, 68%, 143-4.degree. C6H6, 217 m.mu. (log .epsilon. 4.35), inflection point 241 m.mu. (log .epsilon. 4.16), 380 m.mu. (log .epsilon. 3.63), 1515, 1557, 1633 cm.-1 (C: C), 2203 cm.-1 (C:N), 3423 cm.-1 (free NH), 3281 cm.-1 (bound NH); PhCH2, 45%, 152-3.degree., C6H6-cyclohexane, 217 m.mu. (log .epsilon. 4.38), 255 m.mu. (log .epsilon. 4.03), 372 m.mu. (log .epsilon. 3.66), 1508, 1553, 1631 cm.-1 (C:C), 2202 cm.-1 (C:N), 3420 cm.-1 (free NH), 3283 cm.-1 (bound NH). Reaction of I with alkyllithium compds. gave identical substances as above in lower yields. Oxidn. of II (R = Bu) and II (R = Ph), by treating 0.02 mole in 2.3 ml. AcOH in 10 min. with 300 mg. NANO2, keeping the mixt. overnight, and working up as usual gave, resp., 260 mg. III (R = Bu), m. 42-43.degree., .nu.CHCl3max 1459, 1548, 1572 cm.-1 (C:C, C:N), 2234 cm.-1 (C:N), and 400 mg. III (R = Ph), m. 173-3.5.degree. (EtOH-H2O), .nu.(CHCl3) 1443, 1542, 1570 cm.-1 (C:C, C:N), 2235 cm.-1 (C:N). Analogous oxidn. of II (R = PhCH2) (2 g.) gave a mixt. which was sepd. to yield BzH, I, and 410 mg. III (R = PhCH2), m. 86-7.degree. (Me2CO-H2O 1:1), .nu.(CHCl3) 1452, 1491, 1548, 1569 cm.-1 (C:C, C:N), 2234 cm.-1 (C:N). III (R = PhCH2) resisted attempts at further oxidn. with NaNO2 in ACOH.

L9 ANSWER 37 OF 70 CAPLUS COPYRIGHT 2003 ACS on STN

AN 1965:91059 CAPLUS

DN 62:91059

OREF 62:16285h,16286a

TI Organic peroxides. VIII. Reactions of dibenzoyl peroxide and tert-butyl perbenzoate with Grignard reagents and phenyl-lithium

AU Edward, J. T.; Samad, S. A.

CS McGill Univ., Montreal, Can.

SO Pakistan Journal of Scientific and Industrial Research (1964), 7(3), 200-2 CODEN: PSIRAA; ISSN: 0030-9885

DT Journal

LA English

AB cf. CA 62, 506g. Dibenzoyl peroxide was allowed to react with several Grignard reagents under N, in ether at 0-4.degree.. After 30-60 min. stirring the reaction mixt. was treated with cold water, acidified with dil. HCl, and the reaction products sepd. The yields of ester upon reaction of dibenzoyl peroxide with RMgBr were (R and % yield given): p-biphenyl, 15; Bu, 20; Ph, 23; and cydohexyl, 44. Phenyllithium gave poorer yields than the Grignard reagents. tert-Butyl perbenzoate was similar to dibenzoyl peroxide.

- L9 ANSWER 38 OF 70 CAPLUS COPYRIGHT 2003 ACS on STN
- AN 1964:483858 CAPLUS
- DN 61:83858
- OREF 61:14557b-d
- TI The action of anhydrous cobalt chloride and anhydrous uranyl nitrate on several aromatic organomagnesiums and organolithiums. Coupling reactions.
- AU Morizur, Jean Pierre
- CS Ecole Natl. Super. Chim., Paris
- SO Bulletin de la Societe Chimique de France (1964), (6), 1331-7 CODEN: BSCFAS; ISSN: 0037-8968
- DT Journal
- LA Unavailable
- The action of small amts. of anhyd. COCl2 on various Grignard reagents AB (RMqBr) in the presence of PhBr or BuBr gave the dimeric species R2. Compds. prepd. in this way were 55% 2,2'-dimethylbibenzyl; 53% 2,3-dibenzylbutane, b0.07 110.degree.; 55% 2,5-diphenylhexane; 50% 4,4'-diisopropylbiphenyl (II), 60% 4,4'-di(tert-butyl)biphenyl (III); 55% 1,1,2,2-tetraphenylethane (IV) 2,2'-dimethoxy-5,5'-dimethylbiphenyl (V), and 25% 2,2'bithiophene (VI). 4-H2C:CHCH2C6H4MgBr gave an uncharacterized polymeric product. Neither PhMgBr nor 4-BrC6H4MgBr gave the desired dimer. The latter gave 4-BrC6H4Bu with BuBr. Analogous dimerization reactions were observed with various organolithium (RLi) compds. and COCl2 in the presence of PhBr. Compds. prepd. in this way were 65% 2,2'-dimethylbiphenyl, 60% 4,4'-dimethylbiphenyl, 60% I, 60% II, 60% III, 60% IV, 55% 4,4'-dimethoxybiphenyl, 65% V, 65% 4,4'-(N,Ndimethylamino)biphenyl, and 30% VI. All of the coupling reactions are vigorously exothermic. Mechanisms for the reactions are suggested. The reaction of anhyd. UO2(NO3)2 with PhMgBr or PhLi proceeds vigorously to give C6H6, Ph2 (.apprx.25%), and a ppt. of UO3.x-H2O where x = 0.5-2.0.
- L9 ANSWER 39 OF 70 CAPLUS COPYRIGHT 2003 ACS on STN
- AN . 1964:468691 CAPLUS
- DN 61:68691
- OREF 61:11874a-b
- TI Steric difference between the substitution reaction products of lithium alkyls and Grignard reagents with .alpha.-aminonitriles.

 An asymmetric reproduction
- AU Yoshimura, Juji; Ogo, Yoshiaki; Sato, Tetsuo
- CS Inst. Technol., Tokyo
- SO Journal of the American Chemical Society (1964), 86(18), 3858-62 CODEN: JACSAT; ISSN: 0002-7863
- DT Journal
- LA Unavailable
- AB Substitution reactions of 3,4-O-iso-propylidene-N-substituted-D-threosaminonitriles and -D-erythrosaminonitriles with PhMgBr and PhLi have been investigated. The reaction with PhMgBr gave an erythro deriv. and the reaction with PhLi gave threo products predominantly, regardless of the configuration of the .alpha.-carbon in the substrate. N-Substituted acetone-D-glyceroldimines also gave the same result. Consideration of the steric difference between the **Grignard reaction** and the PhLi reaction leads to two possible explanations for the asym. induction of optically active imine-bearing oxygenlike atoms on the .alpha.- and .beta.-carbon.
- L9 ANSWER 40 OF 70 CAPLUS COPYRIGHT 2003 ACS on STN
- AN 1964:60729 CAPLUS

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DN 60:60729
OREF 60:10625c-d
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- TI Halogen-metal interchange reactions of 3,3,3-trichloro-1,2- epoxypropane and of chloral with organolithium compounds and Grignard reagents
- AU Reeve, Wilkins; Fine, Leonard W.
- CS Univ. of Maryland, College Park
- SO Journal of the American Chemical Society (1964), 86(5), 880-2 CODEN: JACSAT; ISSN: 0002-7863
- DT Journal
- LA Unavailable
- AB 3,3,3-Trichloro-1,2-epoxypropane reacts with MeLi or PhLi to form 3,3-dichloroallyl alc. and Me Cl or PhCl. The formation of these products shows that a halogen-metal interchange reaction has occurred. None of the products expected from the opening of the epoxide ring by a carbanion could be detected. Even with BuMgCl and with Bu2Mg, a similar halogen-metal interchange reaction occurs. This is believed to be the first case reported in which a Grignard reagent undergoes a halogen-metal interchange reaction rapidly and to the exclusion of the usually observed reaction path. With BuMgCl, the epoxide ring is also attacked by chloride ion, but not by the Bu carbanion, and the chlorohydrin is formed. PhLi also undergoes a halogen-metal interchange reaction with chloral rather than adding to the carbonyl group in the expected manner. The above reactions indicate that the Cl3C group is unusually reactive in the halogen-metal interchange reaction.
- L9 ANSWER 41 OF 70 CAPLUS COPYRIGHT 2003 ACS on STN
- AN 1963:2894 CAPLUS
- DN 58:2894
- OREF 58:432d-e
- TI Allylic carbanion reactions
- AU Young, William G.
- CS Univ. of California, Los Angeles
- SO Am. Chem. Soc. Div., Petrol. Chem., Preprints (1959), 4(No. 4), B45-B46
- DT Journal
- LA Unavailable
- AB Both MeCH:CHCH2Cl and CH2: CHCHClMe react with Mg to form MeCH:CHCH2MgCl, which, however, reacts with CO2 to form HO2CCHMeCH:CH2. Similarly, PhCH:CHCH2MgBr (colorless) and Me2CO give Me2C(OH)CHPhCH:CH2. This suggests a cyclic mechanism. In contrast, cinnamyllithium, -sodium, or -potassium (highly colored) react with H donors or CO2 to form mixts., predominantly PhCH:CHMe or PhCH:CHCH2CO2H. This suggests a carbanionic mechanism.
- L9 ANSWER 42 OF 70 CAPLUS COPYRIGHT 2003 ACS on STN
- AN 1962:482892 CAPLUS
- DN 57:82892
- OREF 57:16456i,16457a-b
- TI Organic peroxides. III. Reactions of dialkyl peroxides with organolithium and organomagnesium compounds
- AU Baramki, G. A.; Chang, H. S.; Edward, J. T.
- CS McGill Univ., Montreal
- SO Canadian Journal of Chemistry (1962), 40, 441-4 CODEN: CJCHAG; ISSN: 0008-4042
- DT Journal
- LA Unavailable
- AB cf. CA 49, 5379f; 50, 12979a. A series of arylalkyl ethers (I) were prepd. from the reactions of aryllithium (II) or Grignard reagent (III) with dialkyl peroxides (IV). IV dild. with ether was added at

0-80.degree. to ether solns. of II or III. The mixt. was left for 12-14 hrs. and worked up in the usual manner for a **Grignard** reaction. PhOMe was prepd. from di-Me or Me tert-Bu peroxide with PhMgBr or PhLi; PhOEt from di-Et peroxide with PhMgBr or PhLi; tert-BuOPh from ditert-Bu peroxide with PhLi; Me p-anisyl ether from di-Me peroxide with p-anisyllithium or p-anisylmagnesium bromide; Me p-biphenylyl ether from di-Me peroxide with p-biphenylyllithium or p-biphenylylmagnesium bromide; Me 2-naphthyl ether from di-Me peroxide with 2-naphthyllithium; Ph3COMe from di-Me peroxide with Ph3CNa. No tert-BuOPh was obtained from these reactions. The superiority of II than III was observed. Optimum conditions for the reaction were detd. Ionic or 4-center mechanisms are considered for the reactions.

- L9 ANSWER 43 OF 70 CAPLUS COPYRIGHT 2003 ACS on STN
- AN 1962:475578 CAPLUS
- DN 57:75578
- OREF 57:14970f-i
- TI Syntheses of dibiphenylylalkanes, biphenylalkanes and their hydro derivatives with organic magnesium and lithium compounds
- AU Petrov, A. D.; Kaplan, E. P.; Letina, Z. I.
- SO Tr. Vses. Soveshch, po Khim. Pererabotke Neft. Uglevodorodov Poluprod. dlya Sinteza Volokoni Plast. Mass, Baku (1960), 1957, 295-301
- DT Journal
- LA Unavailable
- Reaction of p-PhC6H4MqBr with esters of acetic, butyric, caprylic, AΒ undecylenic, and palmitic acids and hydrogenation of the resulting carbinols with a Cu-Cr catalyst to the corresponding dibiphenylylalkylmethanes, followed by hydrogenation with Raney Ni gave the corresponding bis(bicyclohexyl)alkylmethanes (alkyl groups and m.ps. given): Me 46, Pr 20, C7H15 2, C10-H21 -3, and C15H31 50.degree.. Increasing the length of the alkyl group decreased the viscosity, but increased the oxidn. stability as detd. by the induction period and adsorption of O at 150-200.degree.. Treatment of the di-Li deriv. of biphenyl with various alkyl bromides gave mixts. of the correspond-ing mono-(I) and dialkyldihydrobiphenyls (II) (alkyl substituents and m.ps. given): Bu -1.degree., di-Bu -19.degree., C6H13 7.degree., di-C6H13 -25.degree., C9H19 20.degree., di-C9H19 -12.degree., C10H21 35.degree., di-C10H21 -17.degree.. The viscosities of I were lower than those of II. Stability to oxidn. increased with increasing mol. wt. Comparison of the ultraviolet and infrared spectra of I and II with known hydrogenated biphenyl derivs. indicated that the alkyl group of I was in the 3-position and that the 2 alkyl groups of II were in the 3- and 6-positions.
- L9 ANSWER 44 OF 70 CAPLUS COPYRIGHT 2003 ACS on STN
- AN 1962:403839 CAPLUS
- DN 57:3839
- OREF 57:728d-i,729a-g
- TI Reactions of .alpha.-dimethylaminophenylacetonitrile and its ethylation product with basic or nucleophilic reagents
- AU Morris, Gene F.; Hauser, Charles R.
- CS Duke Univ., Durham, NC
- SO Journal of Organic Chemistry (1962), 27, 465-71 CODEN: JOCEAH; ISSN: 0022-3263
- DT Journal
- LA Unavailable
- AB A study was made of the reactions of .alpha.-(dimethylamino)phenylacetonit rile (I) and its ethylation product (II) with basic or nucleo philic

reagents including KNH2, BuLi, Grignard reagents, LiAlH4, and Na. reactions of I involved ionization of the .alpha.-H, addn. to the nitrile C, and displacement of the nitrile group from the .alpha.-C; those of II occurred at the nitrile C and .alpha.-C. Some interesting comparisons were made be tween the reactions of phenylacetonitrile and I and between those of I and II. I, b0.8 76-8.degree., n25D 1.5116, was prepd. in 94% yield from BzH and the appropriate reagents. I converted into its carbanion by KNH2 in NH3 and this carbanion ethylated with EtBr using 0.8 mole each of the reactants in 800 ml. NH3 gave 137-40 g. II, b0.5 70-2.degree., n25D In an attempt to effect self-condensation of I, a mixt. of 0.10 mole I and 0.05 mole KNH2 in 400 ml. liquid NH3 stirred 8 hrs., replaced by Et20, stirred 14 hrs. at 25-30.degree., 4 ml. AcOH added, followed by H2O, and worked up gave 76% I. BuLi (100~ml.) added to 16.7~g. I in 250~ml. Et2O cooled in an ice bath, after 6 hrs. 13~g. EtBr added, left 1 hr. at room temp., then 250 ml. 1.5N HCl added, refluxed overnight, the 2 layers sepd., the aq. acid layer extd. with Et20, dried, and the residue distd. gave 6 g. propiophenone, b4.2 77-8.degree., n25D 1.5232; 2,4-dinitrophenylhydrazone m. 194-5.degree.. The acid layer made alk. and extd. with Et20 gave 8.9 g. 1-dimethylamino-1-phenyl-2-pentanone (III), b0.3 95-8.degree., n27D 1.5038. Similar results were obtained in other expts. in which the temp. was varied from -80.degree. to 35.degree. and the ratio of propiophenone to III varied considerably. EtMqI (139 ml., 0.793N) added to 16 g. I in 100 ml. Et2O, the mixt. stirred 24 hrs. at room temp., and the product distd. gave 13.8 g. 1-dimethylamino-1phenylpropane (IV), b0.3 41-2.degree., n24D 1.5012; picrate m. 167.5-9.0.degree. The reaction was carried out under several sets of conditions and the following resuits obtained (mole-equivs. EtMgI, temp., time in hrs., % yield of IV, % recovered I given): 0.5, 20.degree., 14, 36, 45; 1.0, 20.degree., 24, 67,n -; 1.0, 35.degree., 2, 48, -; 1.0, 35.degree., 24, 79, 9; 1.1, 35.degree., 24, 85, 0. Recovered I was characterized by acid catalyzed hydrolysis to BzH. BuMgBr (175 ml., 0.677N) added to 17.25 g. I in 250 ml. Et20 at 0.degree., after 48 hrs. the mixt. worked up, and the product distd. gave 16.85 g. ldimethylamino-1-phenylpentane, b0.95 77-8.degree., n18D 1.5002; picrate m. 159-60.degree.. PhMgBr (from 0.2 mole PhBr) in 200 ml. tetrahydrofuran treated with 0.1 mole I in 100 ml. tetrahydrofuran, the mixt. refluxed 5 hrs., decompd., and evapd. gave 22 g. benzhydryldimethylamine-HCl, m. 217-22.degree.. A sample of this salt was hydrolyzed to liberate the free amine, m. 68-70.degree.; MeI salt m. 172-4.degree.. p-Chlorobenzylmagnesium chloride (from p-chlorobenzyl chloride) treated with I gave 1-dimethylamino-1-phenyl-2-(4-chlorophenyl)ethane, b0.57 129-30.degree., n25.5D 1.5644; picrate m. 1345.5.degree.. tert-Butyl magnesium chloride (195 ml., 0.662 N) dild. with 300 ml. Et2O, 16 g. I in 50 ml. Et2O added, after 10 min. 11 g. EtBr added slowly, stirred 1 hr., treated with 100 ml. 2N HCl, the aq. acid layer heated 16 hrs., cooled, extd. with Et2O, dried, and evapd. gave 1.36 g. benzaldehyde 2,4-dinitrophenylhydrazone. No propiophenone was detected. The aq. acid soln. gave 11.1 g. benzyldimethylamine, b15 66-7.degree., n26D 1.4987; picrate m. 93-4.degree.. LiAlH4 (7.6 g.) in 200 ml. Et20 treated with 32 g. I in 100 ml. Et20 to maintain reflux (1260 ml. H evolved), after refluxing 24 hrs. 50 ml. H2O added slowly, the ether sepd., dried, evapd., and the residue distd. gave 17.2 g. material, b0.6 80.degree.. The distillate warmed 24 hrs. with 3N HCl and Et2O gave 14.9 g. 2-dimethylamino-2-phenylethylamine, b0.5 65.degree., n28D 1.5235. I (16 g.) added to 4.6 g. Na in 300 ml. liquid NH3, after 10 min. 16 g. EtI in 100 ml. Et2O added, left 1 hr., decompd., evapd., and the residue distd. gave 7.1 g. benzyldimethylamine, b5 56-8.degree., and 5.05 g. II, b0.4 70-2.degree.; benzyldimethylamine picrate m. 93-4.degree.. In another

expt., in which no EtI was used, was obtained 62% benzyldimethylamine. 2-Morpholino-2-phenylacetonitrile (V) (0.1 mole) in Et2O stirred 16 hrs. with 0.3 mole PhCH2MgCl and the product crystd. gave 20 g. 1-morpholino-1,3-diphenyl-2-propanone, m. 55-60.degree.; HCl salt m. 204-6.degree.. KNH2 (0.2 mole) in 500 ml. liquid NH3 treated 4 hrs. with 37.6 g. II, the product treated with 10.6 g. NH4Cl, evapd., and the product collected gave 22.6 g. 2-dimethylamino-2-phenylbutyramidine (VI), m. 126-7.degree. (C6H6-hexane). Cyclization was effected using 3 g. acetylacetone and 1 g. anhyd. K2CO3 to 6.1 g. VI in 50 ml. alc., refluxing 16 hrs., pouring into 400 ml. H2O, and crystg. to give 2.4 g. 2-(1phenyl-1-dimethylaminopropyl)-4,6-dimethylpyrimidine, m. 223-5.degree.(MeOH). When II was treated with LiNH2 no amidine was isolated and 91% II was recovered. BuLi (125 ml. 1.64N) treated dropwise with 36.2 g. II in 150 ml. Et2O, the soln. left overnight, cooled, treated with H2O, extd. with Et2O, and the residue distd. gave 26 g. 3phenyl-3-dimethylamino-4-octane ketimine (VII), b0.27 114.degree., n24D 1.5218. VII (5.2 g.) heated 20 hrs. with 5% HCl at 50.degree. gave 4.4 g. 3-phenyl-3-dimethylamino4-octanone, b0.3 104-5.degree., n22D 1.5132. PhLi (500 ml. 0.48 N) refluxed 3 hrs. with 45 g. II in 100 ml. Et20, left overnight, and the product distd. gave 44.8 g. 2-dimethylamino-1,2diphenyl-1-butanone ketimine (VIII), b0.3 134-4.degree., m. 76.58.0.degree.. VIII (10 g.) hydrolyzed 16 hrs. with 5% HCl gave 93% 2-dimethylamino-1,2-diphenyl-1-butanone, b0.25 143-4.degree., n26D 1.5784. LiAlH4 (5.9 g.) in 200 ml. Et2O treated dropwise while refluxing with $28.2~\rm g.~II$ in $75~\rm ml.~Et20$, stirred $7~\rm hrs.$, and the mixt. worked up gave $22~\rm g.~1$ -dimethylamino-1-phenylpropane, b0.7 43-4.degree., n23.5D 1.5010; picrate m. 167.5-9.0.degree.. Na (9.2 g.) in 500 ml. liquid NH3 treated with 27.6 g. II, the soln. treated with 100 ml. Me3COH, evapd., the residue treated with 100 ml. H2O and 110 ml. concd. HCl, evapd., cooled, the acid soln. made alk., the mixt. extd. with Et20, dried, the solvent removed, and the residue distd. gave 28.9 g. IV.

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L9 ANSWER 45 OF 70 CAPLUS COPYRIGHT 2003 ACS on STN
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AN 1962:31205 CAPLUS

DN 56:31205

OREF 56:5877f-h

TI A novel type of antioxidant related to polyporic acid

AU Bennett, G. J.; Uri, N.

CS Ministry Agr., Fisheries, and Food, Aberdeen, UK

SO Nature (London, United Kingdom) (1961), 192, 354-5 CODEN: NATUAS; ISSN: 0028-0836

DT Journal

LA Unavailable

AB Compds. related to polyporic acid, 2,5-dihydroxy-3,6-diphenylbenzophenone, contg. hydroxy groups in one of **phenyl** radicals, proved to be very active antioxidants. The reaction of 2,5-dichlorobenzoquinone with the appropriate N-nitrosoacetanilide gave the 3-aryl deriv., which was treated with diazotized 3,4,5-trimethoxyaniline. Hydrolysis of the product then demethylation with HBr-HOAc gave the pentahydroxy quinones.

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L9 ANSWER 46 OF 70 CAPLUS COPYRIGHT 2003 ACS on STN
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AN 1961:75946 CAPLUS

DN 55:75946

OREF 55:14375i,14376a-c

- TI The effect of changing reagent upon stereoselectivity
- AU Stocker, Jack H.; Sidisunthorn, Padet; Benjamin, Ben M.; Collins, Clair J.
- CS Oak Ridge Natl. Lab., Oak Ridge, TN
- SO Journal of the American Chemical Society (1960), 82, 3913-18

CODEN: JACSAT; ISSN: 0002-7863

DT Journal

LA Unavailable

Oxo compds., R1R2CHCOR3, R1R2C(OH)COR3, or R1COCOR2, reacted AB stereoselectively with PhLi, MeLi, Grignard compds., or LiAlH4 to give alcs. with a new asym. center where the ratios threo-erythro or meso-dl were either below or above 1. The reaction of LiAlH4 with 4-methylbenzoin showed only a small stereoselectivity (threo-erythro 1:5.5) as compared with the reaction of PhMgBr with the same compd. (threo-erythro 64:1). The dl-meso ratio was >1 when either PhLi or PhMgI was employed and <1 when PhMgBr or PhMgCl was employed in the addns. of these reagents to biacetyl or phenylacetoin. The ratios of the diastereomers were detd. with the C14-diln. method (CA 46, 4383i; 50, 12884g; 51, 1906b). 1,2-Diphenyl-2-(p-tolyl)ethanol (I) was prepd. by reducing 2.5 g. p-tolyldeoxybenzoin in dry Et20 with 0.3 g. LiAlH4 in Et20. The .alpha.-form (m. 103-4.degree.; acetate m. 107-8.degree.) and the .beta.-form (m. 85-6.degree.; acetate m. 119-20.degree.) of I were sepd. by fractional crystn. from ligroine.

L9 ANSWER 47 OF 70 CAPLUS COPYRIGHT 2003 ACS on STN

AN 1961:37841 CAPLUS

DN 55:37841

OREF 55:7328f-i,7329a-c

- TI Action of Grignard reagents. XX. Action of organomagnesium compounds and of **lithium** aluminum hydride on 3-substituted 3,4-dihydro-4-oxo-1,2,3-benzotriazines
- AU Mustafa, Ahmed; Asker, Wafia; Fleifel, Abdallah M.; Khattab, Samir A.; Sherif, Sayed
- CS Univ. Cairo, Giza
- SO Journal of Organic Chemistry (1960), 25, 1501-3 CODEN: JOCEAH; ISSN: 0022-3263
- DT Journal
- LA Unavailable
- GI For diagram(s), see printed CA Issue.
- AΒ cf. CA 50, 8610b; 54, 24773e. Treatment of C6H4N:N.NR.CO (I) with R'MgX or treatment of the corresponding o-MeO2CC6H4N:NNHR (II) with the same reagent gave the carbinols, o-HOCR2'C6H4N:NNHR (III). PhMgBr (0.9 g. Mg, 9 g. PhBr) in 50 ml. dry Et2O and 1 g. I(R = Ph) or II (R = Ph) heated 3 hrs. on a steam bath and the mixt. kept overnight at 25.degree., and hydrolyzed gave the corresponding III [R, R', % yield from I and II, resp., m.p. (solvent) and color with H2SO4 given]: Ph, Ph, 60, 80, 179-80.degree. (alc.), red; p-MeC6H4, Ph, 85, 87, 154.degree.(alc.), red; p-MeOC6H4, Ph, 87, 90, 132-3.degree. (C6H6-ligroine), red; o-MeOC6H4, Ph, 70, 75, 166.degree. (decompn.) (ligroine b. 90-120.degree.), dark brown; p-BrC6H4, Ph, 85, 90, 130.degree. (C6H6-ligroine), red; p-ClC6H4, Ph, 75, 80, 147.degree. (decompn.) (ligroine), yellowish brown; m-ClC6H4, Ph, 65, 70, 181.degree. (decompn.) (ligroine), dark red; Ph, Me, 72, 80, 140.degree. (alc.), red; p-MeC6H4, Me, 75, 80, 144-5.degree. (ligroine), red; p-MeOC6H4, Me, 65, 70, 125-6.degree. (C6H6-ligroine), red; p-BrC6H4, Me, 80, 90, 135.degree. (C6H6-ligroine), red; p-ClC6H4, Me, 65, 70, 126.degree. (ligroine), yellow. For further study of the effect of the acyl substituted group attached to heterocyclic N compds. with respect to Grignard reagents, the action of PhMqBr on I (R = Ac, Bz) (IV, V) was investigated. V (1 g.) in 40 ml. dry C6H6 treated as above with PhMgBr and the Et2O-C6H6 soln. evapd., the solid residue extd. with 25 ml. cold C6H6 and the insol. residue recrystd. from hot alc. gave 0.42 g. I (R = H) (VI), m. 212-13.degree.. Concn. of the C6H6 ext. and cooling gave 0.39 g. colorless Ph3COH. Similarly, 1 g. IV in 40 ml. C6H6 treated with PhMqBr

in Et2O gave 0.72 g. VI and 0.23 g. PH2MeCOH, m. 82-3.degree., red with H2SO4. LiAlH4 (0.5 g.) in 50 ml. Et2O treated after 15 min. with 1 g. V in 30 ml. C6H6 and the mixt. refluxed 3 hrs., kept overnight at 25.degree. and hydrolyzed with cold dil. HCl gave 0.42 g. VI and PhCH2OH. The stability of VI to LiAlH4 paralleled its behavior toward PhMgBr and III (R = p-MeC6H4, R' = Ph) (VII) was similarly found to be stable. The stability of the N:N system in VII was also observed when 1 g. II (R = p-MeC6H4) in 30 ml. C6H6 was treated as above with 0.5 g. LiAlH4 in 50 ml. Et2O to give colorless o-(p-MeC6H4NHN:N)C6H4CH2OH, identified as 0.24 g. of the corresponding urethan, m. 119-20.degree.. V (1 g.) and 2 g. AlCl3 heated 1 hr. at 120-5.degree. (oil bath) and the cooled mixt. decompd. with 100 ml. ice H2O contg. 5 ml. concd. HCl, the solid washed and dried and recrystd. from alc. gave VI, similarly eliminating the acyl group.

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L9 ANSWER 48 OF 70 CAPLUS COPYRIGHT 2003 ACS on STN
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AN 1960:128408 CAPLUS

DN 54:128408

OREF 54:24479h-i,24480a-b

TI Organo-tin compounds. XII. The action of **lithium** aluminum hydride and Grignard reagents on organo-tin nitriles and esters

AU van der Kerk, G. J. M.; Noltes, J. G.

CS Org. Chem. Inst. T.N.O, Utrecht, Neth.

SO Journal of Applied Chemistry (1959), 9, 176-9 CODEN: JACHAU; ISSN: 0021-8871

DT Journal

LA Unavailable

cf. CA 54, 1379a. The redn. by LiAlH4 in ether of Pr3Sn(CH2)2 CN (I) to (3-aminopropyl)tripropyltin, b12 140-5.degree., and of Bu3Sn(CH2)2CO2Me, Ph3Sn(CH2)3CO2Me, and Ph3Sn(CH2)4CO2Me to (3-hydroxypropyl)tributyltin, b12 117-19.degree., (4-hydroxybutyl)triphenyltin, m. 75-6.degree., and (5-hydroxypentyl)triphenyltin, m. 64-6.degree., resp., occurred without Sn-C bond cleavage in 34-77% yields. MeMgI with I gave 49% of (3-oxobutyl)tripropyltin (I), b0.2 84-9.degree. (2,4-dinitrophenylhydrazone m. 48-50.degree.; oxime b0.2 110.degree.; semicarbazone m. 78-80.degree.), and 11% of Pr3MeSn, but an 89% yield of (3-hydroxy-3-methylbutyl)tributyltin, b0.7 133-8.degree. No Bu3MeSn was obtained from Bu3Sn(CH2)2CO2Me. Similarly prepd. from PhMgBr and nitriles were (2-benzoylethyl)triphenyltin, m. 79-80.degree. (2,4-dinitrophenylhydrazone m. 174-6.degree.), and (2-benzoylethyl)tripropyltin, b0.05-0.30 158-65.degree. (2,4-dinitrophenylhydrazone m. 106.degree.).

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L9 ANSWER 49 OF 70 CAPLUS COPYRIGHT 2003 ACS on STN
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AN 1959:16717 CAPLUS

DN 53:16717

OREF 53:3041c-i,3042a-i,3043a-e

TI Unsaturated aldehydes and ketones. II. A new reaction of magnesium - and lithium-organic compounds

AU Jutz, Christian

CS Tech. Hochschule, Munich, Germany

SO Chemische Berichte (1958), 91, 1867-81 CODEN: CHBEAM; ISSN: 0009-2940

DT Journal

LA Unavailable

AB cf. C.A. 52, 17266a. MePhN(CH:CH)nCHO (I) (n = 1, 2) and MePhNCH:CHCOR (II) (R = alkyl or aryl) with Mg- and Li-org. compds. yield aldehydes and ketones of the type R(CH:CH)nCHO (III) (n = 1, 2) (R = aryl, aralkenyl, alkenyl, alkynyl). The appropriate Grignard soln. filtered through

glass wool added dropwise to the I or II in C6H6-Et2O, the mixt. stirred 0.5 hr. without cooling, treated dropwise with N H2SO4, the aq. layer extd. with Et2O, and the org. solns. combined gave the corresponding III; the aromatic aldehydes were purified through the NaHSO3 adducts and regenerated with Na2CO3 or K2CO3. HC.tplbond.CCHO in EtOH treated with PhNHMe yielded MePhNCH: CHCHO (IV); MePhCH: CHAc (V), b0.5 135.degree. to b0.1 118.degree., was obtained similarly from HC.tplbond.CAc and PhNHMe. The Grignard reagent from 7.8 g. PhBr, 2 g. Mg, and 15 cc. Et20 treated with 8.9 g. IV in 50 cc. C6H6 and 50 cc. Et2O yielded 80% PhCH: CHCHO (VI); phenylhydrazone, lemon-yellow needles, m. 167.5-68.degree. (decompn.); 2,4-dinitrophenylhydrazone, brick-red crystals, m. 253-4.degree. (pyridine-EtOH). PhLi (from 55 g. PhBr and 5.5 g. Li in 250 cc. Et20)(22 cc.) added dropwise with 4 g. IV in 25 cc. C6H6 and 25 cc. Et2O gave 95% VI. The Grignard reagent from 17.2 g. p-MeC6H4Br, 3.5 g. Mg, and 60 cc. Et20 treated with 18 g. IV in 80 cc. C6H6 and 80 cc. Et20 and the crude product distd. and purified through the NaHSO3 adduct gave 11.5 g. p-MeCH: CHCHO, leaflets, m. 42-3.degree. (ag. MeOH). p-MeOC6H4Br (9.5 g.) treated with 2 g. Mg in 10 cc. tetrahydrofuran and 30 cc. Et20, the mixt. treated with 10 q. IV in 50 cc. C6H6 and 70 cc. Et2O, the crude product purified through the NaHSO3 adduct, sublimed, and recrystd. from hexane yielded 4.5 g. p-MeOC6H4CH:CHCHO, leaflets, m. 59.degree.. The Grignard reagent from 12 g. p-PhC6H4Br, 2 g. Mg, 30 cc. tetrahydrofuran, and 10 cc. Et20 treated with 10 g. IV in 50 cc. C6H6 and 70 cc. Et20 yielded 6.5 g. p-PhC6H4CH: CHCHO, pale yellow needles, m. 120-1.degree. (hexane and MeOH). Ph2C:CHBr (VII) (8 g.) treated with 1.5 g. Mg in 20 cc. tetrahydrofuran, the resulting deep green Grignard soln. treated with 6 g. I in 30 cc. C6H6 and 40 cc. Et20, and worked up in the usual manner yielded 4-5 g. Ph2C:CHCH:CHCHO, pale yellow needles, m. 69.5-70.degree. (aq. MeOH); 2,4-dinitrophenylhydrazone, deep violet-red needles, m. 249-51.degree. (PhMe-EtOH). PhCH:CHBr (9.5 g.) treated with 2 g. Mg and 20 cc. tetrahydrofuran and the mixt. treated with 10 g. IV yielded 5 g. Ph(CH:CH)2CHO (VIII), m. about 16.degree.; p-nitrophenylhydrazone, red-golden leaflets, m. 206-7.degree. (MeOH); phenylhydrazone, yellow needles, m. 178-9.degree. (decompn.); anil, yellow needles, m. 108-10.degree. (decompn.). For purification the crude VIII in Et2O was shaken with aq. NaHSO3 to yield the adduct; the Et2O layer worked up gave in several runs up to 2 g. (PhCH:CH)2, leaflets, m. 152.degree... Grignard reagent from 11.2 g. EtBr, 3 g. Mg, and 15 cc. Et20 treated with 10.2 g. PhC.tplbond.CH in 50 cc. C6H6, refluxed 2 hrs., treated with 18 g. IV in 80 cc. C6H6 and 80 cc. Et2O, concd., the concentrate shaken with 20 cc. aq. NaHSO3, and the adduct decompd. and processed in the usual manner yielded PhC.tplbond.CCH:CHCHO (IX), b0.2 about 110.degree.; p-nitrophenylhydrazone, yellow needles, m. 205.degree. (decompn.) (MeOH). EtBr (5.6 g.) treated with 2 g. Mg and 10 cc. Et2O, the resulting EtMgBr treated with 6.6 g. p-MeOC6H4C.tplbond.CH in 25 cc. C6H6, heated to boiling, distd. to remove 10-15 cc. solvent mixt., the mixt. refluxed 2 hrs. and treated with 10 q. IV in 40 cc. C6H6 and 40 cc. Et2O, and the product purified through the NaHSO3 adduct yielded about 2.8-3.5 g. p-MeO deriv. of IX, needles or leaflets, m. 49-50.degree. (hexane and aq. MeOH); phenylhydrazone, pale yellow, light-sensitive leaflets, m. 129-30.degree. p-nitrophenylhydrazone, orange-red crystals, m. 173-4.degree. (decompn.)(C6H6-ligroine). The Grignard reagent from 20.7 g. 1-C10H7Br, 3 g. Mg, 10 cc. tetrahydrofuran, and 20 cc. Et2O treated with 18 g. IV in 80 cc. C6H6 and 80 cc. Et2O, and the crude product purified gave 11-12 g. 1-C10H7CH:CHCHO (X), pale yellow needles, m. 47-8.degree. (hexane), b0.4 about 160-70.degree.; oxime, needles, m. 165-6.degree. (decompn.); phenylhydrazone, yellow leaflets, m. 125-6.degree. (decompn.) (hexane and aq. MeOH). The Grignard reagent from 10.4 g. 2-C10H7Br, 2 g. Mg, 10 cc.

tetrahydrofuran, and 10 cc. Et20 with 10 g. IV in 50 cc. C6H6 and 50 cc. Et2O yielded 6 g. 2-isomer of X, needles, m. 125.degree. (hexane and MeOH); 2,4-dinitrophenylhydrazone, red crystals, m. 254-5.degree. (decompn.) (PhMe). The Grignard reagent from 8.2 g. .alpha.bromothiophene, 2 g. Mg, 15 cc. tetrahydrofuran, and 10 cc. Et2O yielded with 10 g. IV in 50 cc. C6H6 and 50 cc. Et2O 4-4.5 g. 1-(2-thienyl)-1propen-3-al, b2.0 101-2.degree., n20D 1.68, turning dark brown and forming a resin in a few days; 2,4-dinitrophenylhydrazone, deep red leaflets and prisms, m. 249.degree. (decompn.) (glacial AcOH); semicarbazone, m. 219-21.degree. (decompn.) (EtOH). The Grignard reagent from 4 g. PhBr, 1 g. Mg, and 10 cc. Et2O treated with 5.2 g. MePhN(CH:CH)2CHO (XI) in 80 cc. C6H6 and 50 cc. Et2O yielded through the NaHSO3 adduct 2 g. VIII, pale yellow oil. The Grignard reagent from 8.7 g. p-MeC6H4Br, 2 g. Mg, and 15 cc. Et2O with 10 g. XI in 150 cc. C6H6 and 50 cc. Et2O gave 3-5 g. p-Me deriv. of VIII, pale yellow leaflets, m. 102-3.degree. (hexane). The Grignard reagent from 4.8 g. p-MeOC6H4Br, 1.5 g. Mg, 20 cc. tetrahydrofuran, and 100 cc. Et20 with 5.2 q. XI in 80 cc. C6H6 and 50 cc. Et20 yielded 1.8-2.2 q. p-MeO deriv. of VIII, pale yellow needles or leaflets, m. 77-8.degree. (MeOH), intensely yellow in MeOH soln. Grignard reagent from 6.5 g. VII, 1.5 g. Mg, and 20 cc. tetrahydrofuran treated with 5.2 g. XI in 80 cc. C6H6 and 50 cc. Et2O yielded 2-2.5 g. Ph2C:CH(CH:CH)2CHO, lemon-yellow needles, m. 101.degree. (aq. MeOH). The Grignard reagent from 15.8 g. PhBr, 3 g. Mg, and 30 cc. Et2O treated with 18 g. V in 150 cc. Et20 and distd. gave 8.7 g. PhCH: CHAc, b16 139-41.degree., prisms, m. 41.degree. (hexane and ag. MeOH); 2,4-dinitrophenylhydrazone, brick-red leaflets and needles, m. 220-2.degree. (PhMe). The Grignard reagent from 9.2 g. PhCH:CHCHO, 2 g. Mg, and 20 cc. tetrahydrofuran treated with 10 g. V in 150 cc. Et2O, the crude product distd., the distillate (7 g.), b0.5 135-40.degree. to b1.2 155.degree., dissolved in 15 cc. MeOH, and the soln. dild. gradually with H2O gave Ph(CH:CH)2CHO, pale yellow needles, m. 66-7.degree. (aq. MeOH and hexane); semicarbazone, pale yellow, light-sensitive needles, m. 192-4.degree. (decompn.) (MeOH and C6H6). The Grignard reagent from 11.2 g. EtBr, 3 g. Mg, and 15 cc. Et20 treated dropwise with 10.2 g. PhC.tplbond.CH in 50 cc. C6H6, refluxed 12 hrs., treated with 19 g. V in 180 cc. Et2O, worked up, and the crude oily product distd. yielded 14.6 g. PhC.tplbond.CCH:CHAc (XI), needles, m. 42-3.degree., b0.01 96.degree.. XI (2.420 g.) hydrogenated in 75 cc. MeOH over 80 mg. PtO2 during 0.75 hr., filtered, and evapd. in vacuo yielded 2.25 g. Ph(CH2)2Ac, b10 97-7.5.degree., n18D 1.5058; semicarbazone, needles and leaflets, m. 143-4.degree. (aq. MeOH and hexane). MeBr passed into 30 cc. Et2O contg. 1.2 g. Mg until all Mg had been dissolved, the soln. refluxed 0.5 hr., treated with 9 g. IV in 80 cc. C6H6 and 80 cc. Et2O, the Et2O soln. of the product treated with 5 g. H2NCONHNH2.HCl and 7.5 g. NaOAc in concd. aq. soln., the org. solvents distd. in vacuo, and the residue dild. with H2O and filtered yielded a small amt. H2NCONHN: CHCH: CH2, m. 198-201.degree. (decompn.). The Grignard reagent from 5.5 g. EtBr, 2 g. Mg, and 20 cc. Et2O treated with 9 g. IV in 80 cc. C6H6 and 80 cc. Et2O gave 0.6-0.7 g. EtCH: CHCHO, b80 62-4.degree., n22D 1.4469; semicarbazone, leaflets and needles, m. 175-7.degree. (decompn.) (aq. MeOH). The Grignard reagent from 6.2 g. iso-PrBr, 2 g. Mg, and 20 cc. Et2O treated with 9 g. IV in 80 cc. C6H6 and 80 cc. Et2O gave 1.4-1.5 iso-PrCH:CHCHO, b48 59-60.degree., n24D 1.4396; semicarbazone, leaflets, m. 174-5.degree. (decompn.) (MeOH). The Grignard reagent from 12 g. Me3CCl, 3 g. Mg, 30 cc. tetrahydrofuran, and 20 cc. Et20 treated with 18 g. IV in 80 cc. C6H6 and 80 cc. Et20 yielded 2.8 g. Me3CCH:CHCHO, b50 72-3.degree., n24D 1.4387; 2,4-dinitrophenylhydrazone, red leaflets, m. 229.degree. (C6H6-EtOH); semicarbazone, leaflets, m. 169-70.degree. (MeOH). The Grignard reagent

from 6.2 g. PrBr, 2 g. Mg, and 20 cc. Et20 treated with 9 g. IV in 80 cc. C6H6 and 80 cc. Et2O gave 1.5-2.0 g. PrCH:CHCHO, b50 70-2.degree., n24D 1.4441; semicarbazone, leaflets, m. 169-71.degree. (decompn.) (MeOH); p-nitrophenylhydrazone, golden-orange leaflets, m. 134-5.5.degree. (MeOH). The Grignard reagent from 6.9 g. iso-BuBr, 2 g. Mg, and 20 cc. Et2O with 9 q. IV in 80 cc. C6H6 and 80 cc. Et2O gave 1.7-2.2 g. iso-BuCH: CHCHO, b50 81-3.degree., n23.5D 1.4412; semicarbazone, m. 181-3.degree. (decompn.) (MeOH); 2,4-dinitrophenylhydrazone, brick-red leaflets, m. 149-50.degree. (decompn.) (dioxane). The Grignard reagent from 6.9 g. BuBr, 2 g. Mg, and 20 cc. Et2O with 9 g. IV in 80 cc. C6H6 and 80 cc. Et2O gave 1.7 g. BuCH: CHCHO, b50 90-1.degree., n19D 1.4486; semicarbazone, leaflets, m. 167.degree. (decompn.) (MeOH); p-nitrophenylhydrazone, golden-orange leaflets, m. 118-19.degree. (MeOH). The Grignard reagent from 7.6 g. AmBr with 9 g. IV gave in the usual manner 2.2-2.5 g. AmCH:CHCHO, b9 69-71.degree., n20D 1.4470. C7H15MgBr from 9 g. C7H15Br and 9 g. IV yielded similarly 2.7-3.2 g. C7H15CH:CHCHO, b1.5 72-3.degree., n2OD 1.4470; semicarbazone, leaflets, m. 163-5.degree. (decompn.) (C6H6-ligroine); 2,4-dinitrophenylhydrazone, brick-red leaflets, m. 124-5.degree. (ligroine, b. 80-120.degree.). EtMgBr from 5.7 g. EtBr in 10 cc. Et20 treated dropwise with 4.1 g. BuC.tplbond.CH in 50 cc. C6H6, refluxed 2 hrs., treated with 9 g. IV in 80 cc. C6H6 and 80 cc. Et2O, and the crude product distd. yielded about 5-5.5 g. BuC.tplbond.CHCH:CHCHO, b10 94-5.degree., n23.5D 1.5110, which turned rapidly yellow-orange and resinified in air and light; semicarbazone, m. 192-4.degree. (decompn.) (MeOH). BuMgBr (13.8 g.) treated with 3 g. Mg and 40 cc. Et2O and the BuMgBr treated with 19 g. V in 150 cc. Et2O yielded 6.8 g. BuCH: CHAc, bl1 69-70.degree., n16D 1.4495; 2,4-dinitrophenylhydrazone, light brick-red needles, m. 94-5.degree. (MeOH); 2,4-nitrophenylhydrazone, orange-yellow prisms, m. 109-10.degree. (aq. MeOH and hexane). EtMgBr from 11 g. EtBr in 15 cc. Et20 treated with 8.2 g. BuC.tplbond.CH in 75 cc. C6H6, refluxed 2 hrs., treated with 18 g. V in 150 cc. Et2O, and worked up yielded 10-11 g. BuC.tplbond.CCH:CHAc (XII), b1.0 70.degree. to b3.0 90.degree., n17D 1.5050; semicarbazone, m. 122-3.degree. (decompn.) (MeOH); p-nitrophenylhydrazone, orange needles, m. 128-9.degree. (C6H6-hexane). XII (3.757 g.) in 75 cc. MeOH hydrogenated 0.75 hr. over 80 mg. PtO2 gave 3.4 g. C8H17Ac, b1.4 62-3.degree., n16.5D 1.4276; 2,4-dinitrophenylhydrazone, dark yellow needles, m. 73-4.degree. (MeOH); p-nitrophenylhydrazone, yellow prisms, m. 97-8.degree. (aq. MeOH); semicarbazone, leaflets, m. 125-6.degree. (MeOH).

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L9 ANSWER 50 OF 70 CAPLUS COPYRIGHT 2003 ACS on STN
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AN 1959:7033 CAPLUS

DN 53:7033

OREF 53:1327c-i,1328a-d

TI Reactions of some ester alkaloids and related synthetic compounds with the phenvl Grignard reagent

AU Zaugg, Harold E.; Michaels, Raymond J.; DeNet, Robert W.

CS Abbott Labs., N. Chicago

SO Journal of Organic Chemistry (1958), 23, 847-51 CODEN: JOCEAH; ISSN: 0022-3263

DT Journal

LA Unavailable

AB Reactions of arecoline (I), cocaine (II), anhydroecgonine Me ester (III), and pilocarpine (IV) with PhMgBr (V) were carried out and the products further transformed. In addn., the 2 stereoismoeric 3-acetyl-1,4-dimethyl-4-hydroxypiperidines (VI) and 3 simple imidazole esters, 4-carbomethoxyimidazole (VIII), 4-carbethoxy-5-methylimidazole (VIII), and 4-carbomethoxy-2-methylimidazole (IX) were treated similarly. The

stereochem. of several of the products was discussed and their preliminary pharmacol. assay is reported. V (43 g.) in 300 ml. Et20 treated portionwise with 15 g. I. HBr, the mixt. refluxed 2 hrs., left overnight, cooled, and treated dropwise with aq. NH4Cl, the Et2O layer sepd., extd. with dil. HCl, the acid extd. cooled, made alk., extd. with Et2O, dried, and distd. gave 6 g. cis-ketone (X), m. 115-16.degree.; MeI deriv., m. 217-18.degree. (decompn.) (aq. alc.). X (10 g.) and 60 ml. 48% aq. HBr refluxed overnight, poured into H2O, made alk., the oil taken up in Et2O, and the soln. distd. gave 7.2 g. trans-X (XI), m. 62-3.degree.(hexane): HCl salt, m. 230-1.degree. (decompn.) (alc.-Et20). MeMgI (from 5.7 g. MeI and 1 g. Mg in 50 ml. Et20) treated rapidly with 7 g. X in 50 ml. C6H6, refluxed 2 hrs., and isolated gave 5.3 g. cis-N-methyl-3-(.alpha.-hydroxy-.alpha.-phenethyl)-4-phenylpiperidine (XII), m. 86-7.degree. (hexane). Similarly, XI gave 84% trans-isomer (XIII), m. 142-3.degree.(cyclohexane). V (0.7 mole) in 600 ml. Et2O refluxed 18 hrs. with 0.088 mole II in 500 ml. Et20 and worked up gave 15.4 q. glycol (XIV), m. 185-6.degree. (alc.), [.alpha.]27D -23.5.degree. (0.04 g./ml. CHCl3), pKa 7.49. XIV in MeCOEt treated 1 hr. at 40.degree. with a slight excess of Me2SO4, Et2O added, and cooled gave the quaternary methomethyl sulfate. XIV (8.4 g.), 18 ml. concd. HCl, and 60 ml. AcOH refluxed 2 hrs., concd. to dryness, the residue dissolved in H2O, made strongly alk., the pptd. oil taken up in Et20, dried, and concd. gave 4.5 g. diene (XV), b2.5 201-3.degree., n25D 1.6338, [.alpha.]30D 548.degree. (c 0.04, CHCl3), .lambda. 282 m.mu., .epsilon. 17,800; MeI deriv., m. 281-2.degree. (alc.), [.alpha.]28D 450.degree. (c 0.004, H2O). Anhydroecgonine-HCl (13 g.) 6.5 g. Amberlite XE-156 resin, 50 ml. MeOH, and 175 ml. C2H4Cl2 refluxed 21 hrs., the mixt. filtered, the filtrate concd. to dryness, the residual glassy HCl salt treated with sufficient satd. K2CO3 to produce a fluid mixt., treated with solid anhyd. K2CO3 to further salt out the base, the product taken up in Et20, and isolated gave 7.1 g. III, b10 124-6.degree., n25D 1.5006. PhLi (prepd. by adding dropwise 42.4 g. PhBr in 200 ml. Et2O to 3.6 g. Li in 100 ml. Et2O and refluxing 2 hrs.) heated and stirred 1 hr. with 8.1 g. III in 100 ml. dry Et20, cooled, decompd., and the solids collected gave 7.7 g. diphenylcarbinol (XVI), m. 209-10.degree. (aq. MeOH), [.alpha.]25D -52.degree. (c 0.011, CHCl3), the infrared spectrum showed the typical OH but no CO absorption. XVI (1.5 g.) with 0.6 g. Me2SO4 gave 1.1 g. methomethyl sulfate, m. 186-7.degree.. Dehydration of 1.5 q. XVI with 4 ml. concd. HCl and 12 ml. AcOH gave crude XV, converted directly to the methiodide. Further proof of XV prepd. by the above two methods comes from the fact that their infrared spectra in CHCl3 were qualitatively identical and their specific rotations differed by only 2%. IV (10 g.) in dry C6H6 similarly refluxed overnight with V and isolated gave a mixt. of .alpha. - and .beta. -glycols (XVII); from the .alpha. -form of IV, m. 130.degree., was obtained 45% .alpha.-form of XVII, m. 177-8.degree. (EtOAc). From the .beta.-form of IV was obtained 11% .beta.-form of XVII, m. 180-1.degree. (decompn.) (EtOAc). An attempt to increase the yield of the .beta.-glycol of XVII by using PhMe instead of C6H6 in the Grignard reaction gave the same results. The lower reactivity of the .beta.-form as compared to the .alpha.-isomer was further indicated by the fact that the initial stage of the Grignard reaction of the .alpha.-form is exothermic. The .beta.-form showed no such evidence of spontaneous reaction. V (from 37.7 g. PhBr) treated portionwise with 15 g. powd. IV.HCl, refluxed overnight, decompd. with satd. aq. NH4Cl, the free base collected, and recrystd. gave 14.3 g. glycol (XVIII), m. 291-3.degree. (decompn.) (HCONMe); HCl salt. m. 137-9.degree.(decompn.), [.alpha.]27D -145.degree. (c 0.008, H2O). XVIII (3.6 g.) in 30 ml. AcOH contg. 10 ml. concd. HCl refluxed 2 hrs., concd., the residue dissolved in H2O, the soln. made

alk., extd. with Et2O, dried, concd., and the oil treated with Et2O-HCl gave 2 g. tetrahydrofuran compd. (XIX). HCl, m. 254-6.degree. (alc.-Et20), [.alpha.]27D 156.degree. (c 0.01, H2O). IV (54 g.) in 300 ml. Et2O treated with 250 ml. tetrahydrofuran, the Et20 removed, the hot Grignard reagent treated with 10 g. VIII, refluxed a few min., most of the solvent removed, Et2O added to the cold soln., the solid collected, the Et2O layer sepd., concd., the residue combined with the original filter cake, dissolved in excess HCl, the soln. made alk., and the 11.5 g. of the liberated base again collected gave on recrystn. the corresponding carbinol (XX), m. 186-7.degree. (decompn.) (iso-PrOH). Refluxing 1.5 g. XX, MeCOEt, and excess MeI 4 hrs. gave 0.8 g. quaternary methiodide of the N-Me deriv. of XX, m. 223-4.degree. (decompn.) (alc.). Similarly, addn. of IV to VII gave 81% carbinol, m. 173-4.degree.(decompn.), and reaction of IX with IV led to 78% carbinol, m. 200-1.degree. (decompn.) (alc.).

ANSWER 51 OF 70 CAPLUS COPYRIGHT 2003 ACS on STN L9

AN 1957:71408 CAPLUS

DN 51:71408

OREF 51:12873a-f

1,2-Addition of allylmagnesium bromide to N-diphenylmethyleneaniline and structurally related systems

ΑU Gilman, Henry; Eisch, John

Iowa State Coll., Ames CS

Journal of the American Chemical Society (1957), 79, 2150-3 SO CODEN: JACSAT; ISSN: 0002-7863

DTJournal

LΑ Unavailable

AΒ Ph2C:NHPh (I) (43.3 g.) in 100 cc. dry Et2O treated during 25 min. with 0.215 mole CH2: CHCH2MgBr (II) in 190 cc. Et2O, refluxed 18 hrs. with stirring, and hydrolyzed with 500 cc. satd. aq. NH4Cl, the Et2O layer worked up, and the crude product (48.1 g.) recrystd. from 200 cc. 95% EtOH yielded 44.0 g. CH2:CHCH2CPh2NHPh (III), m. 78.5-80.degree.. PrLi (0.097 mole) in 90 cc. Et20 added over 45 min. to 20.0 g. I in 50 cc. dry Et20, the mixt. refluxed 6 hrs., stirred overnight, and treated with H2O, the org. layer worked up, and the residue (21.2 g.) recrystd. from 100 cc. 95% EtOH yielded 18.0 g. Ph2PrCNHPh (IV), m. 85-5.5.degree. (from EtOH). III (3.0 g.) in 50 cc. EtOAc hydrogenated over 30 mg. prereduced PtO2 in 50 cc. 95% EtOH, filtered, concd., and refrigerated gave 2.5 g. IV, m. 83-5.degree.. PhLi (0.144 mole from 0.145 mole PhBr and 2.2 g. Na) in 100 cc. dry Et2O treated during 50 min. with 15.5 g. PhPrC:NPh (V) in 75 cc. dry Et20, stirred 4 hrs., and hydrolyzed, and the crude, somewhat sticky solid (22 g.) recrystd. from 100 cc. 95% EtOH yielded 12.4 g. IV, m. 83-4.degree.. Filtered PrMgBr (0.60 mole) in 600 cc. Et20 treated during 45 min. with 50.0 g. PhCN, refluxed overnight, poured into 200 cc. 6N H2SO4, heated to remove the Et2O, cooled, and extd. with Et2O, and the ext. distd. yielded 64.0 g. BzPr, bl3 106.5-107.degree.. PhNH2 (25 cc.) added to 25.0 g. ZnCl2.2H2O in 40 cc. H2O, 20 cc. EtOH, and 10 cc. concd. HCl, the mixt. stirred overnight and filtered, and the solid residue washed twice with Et2O and dried yielded 30.0 g. PhNH2-ZnCl2 complex (VI). Powd. VI (2 g.) added to 30 cc. PhNH2 and 30.0 g. BzPr at 100.degree., the mixt. heated during 15 min. slowly to 160.degree., cooled, dild. with 300 cc. CHCl3, and filtered, and the filtrate worked up yielded 15.8 g. V, b13 183-5.degree., bl.1 128.degree., nD25 1.5926. Fluorenone anil (25.5 g.) in 100 cc. dry Et20 treated during 20 min. with an equimolar amt. of II in 110 cc. Et2O and the mixt. stirred 2 hrs. and hydrolyzed with aq. NH4Cl gave 25.7 g. .alpha.-allylfluorenylaniline, long needles, m. 136.5-7.5.degree. (from EtOH with Norit). 6-Phenylphenanthridine (25.5 g.) in 150 cc. dry Et2O treated during 0.5 hr. with 0.125 mole II in 125

9/24/2003> Patel

cc. Et20, refluxed overnight, cooled, and hydrolyzed, and the crude white product (28.1 g.) repeatedly recrystd. from 95% Et0H yielded 20 g. 6-allyl-6-phenyl-5,6-dihydrophenanthridine, platelets, m. 103-4.5.degree.. I (18.0 g.) in 100 cc. dry Et20 heated 18 hrs. with 0.01 mole MeMgI or PrMgBr in Et20 gave only about 16-17 g. unchanged I.

- L9 ANSWER 52 OF 70 CAPLUS COPYRIGHT 2003 ACS on STN
- AN 1957:46878 CAPLUS
- DN 51:46878
- OREF 51:8681d-i,8682a-i
- TI Chemistry of acetylenic ethers. XIX. The reaction of aldehydes with ethoxyethynylmagnesium bromide
- AU Postma, J. C. W.; Arens, J. F.
- CS Univ. Groningen, Neth.
- SO Rec. trav. chim. (1956), 75, 1385-96
- DT Journal
- LA English
- Aldehydes with BrMqC.tplbond.COEt (I) do not yield the expected secondary AB carbinols. In the case of aromatic aldehydes the products are formed from 2 mols. of the aldehyde and 1 mol. of the acetylenic compd. structures of these compds. have been established by several transformations and the chemistry of their formation has been studied. Aliphatic aldehydes and I give products derived from 3 mols. of the aldehyde and 1 of the acetylenic compd. I was prepd. by slowly adding 16 g. HC.tplbond.COEt in 150 ml. abs. Et20 to a stirred soln. of 0.21 mole EtMgBr in 250 ml. Et20, when the evolution of C2H6 ceased, the mixt. was cooled in ice, treated dropwise with 32 g. BzH in 100 ml. abs. Et20, refluxed 10 min., hydrolyzed with 70 g. NH4Cl in 300 ml. H2O, and the Et2O layer sepd., washed with H2O, dried with Na2SO4, and evapd. in vacuo under N at 30 degree.; the resulting crystals, filtered and washed with ice-cold EtOH, yielded 29-35% PhCH(OH)C(:CHPh)CO2Et (II), m. 90-1 degree. (from EtOH); a better yield (51%) was obtained by hydrolyzing the reaction mixt. with 150 ml. 2N H2SO4 instead of NH4Cl. BzH and LiC.tplbond.COEt gave PhCH(OH)C.tplbond.COEt (III). III (4.3 g.) in 20 ml. Et2O and 0.03 mole EtMgBr in 25 ml. Et2O refluxed until the evolution of C2H6 ceased, 2.5 g. BzH in 20 ml. Et2O added, the mixt. refluxed 15 min., hydrolyzed with 10 g. NH4Cl in 50 ml. H2O, the Et2O layer washed with H2O, dried with Na2SO4, evapd. in vacuo and the resulting solid filtered off and washed with ice-cold EtOH yielded 3.5 q. II, m. 91-2.degree. (from EtOH). II (7 g.) and 0.5 q. p-MeC6H4SO3H in 100 ml. PhMe refluxed 2 hrs., the cooled mixt. washed with Na2CO3 soln. and H2O, dried over Na2SO4, evapd. in vacuo, and the resulting oil crystd. yielded 5.2 g. Et 3-phenyl -2-indenecarboxylate (IV), m. 90.5-1.5.degree. (from petr. ether). IV (3 g.) and 40 ml. aq. HI (azeotrope, b. 125-7.degree.) refluxed 1 hr., cooled, the ppt. filtered off, washed with 50% aq. alc., and the remaining crystals dissolved in 2N NaOH, repptd. with HCl, sublimed in vacuo, and recrystd. from AcOH yielded 1.7 g. 3-phenyl-2-indenecarboxylic acid (V), yellow needles, m. 216-17.degree., also formed by sapon. of IV with hot KOH in MeOH. II (1 g.) refluxed with 20 ml. 2N KOH in MeOH, the soln. evapd. in vacuo, the residue dissolved in H2O, the soln. extd. with Et20 to remove unsapond. material, the aq. layer acidified, and the ppt. filtered off and crystd. from dil. AcOH yielded 700 mg. PhCH(OH)C(:CHPh)CO2H, colorless crystals, m. 158.5-9.0.degree. (decompn.). II (1 g.) in 100 ml. dry Me2CO treated with 2.5 g. KMnO4 in small portions at 30.degree., the soln. stirred 16 hrs. at room temp.; refluxed 3 addnl. hrs., the brown ppt. filtered off, washed with dry Me2CO, the solid suspended in H2O, and SO2 passed into the soln. yielded 76% BzOH. V similarly oxidized yielded 60% o-BzC6H4CO2H, m. 128-9.degree.. II (1 g.)

in 30 ml. Me2CO at 0-5.degree. treated dropwise with 0.35 g. CrO3 in 0.3 ml. concd. H2SO4 and 3 ml. H2O, stirring continued 1.5 hrs., 200 ml. H2O added, and the ppt. filtered off and crystd. from Et2O yielded 0.8 g. PhCH:CBzCO2Et, m. 95.0-6.5.degree.. V (200 mg.) in 10 ml. 2N aq. NaOH and 1 g. powd. 8% Na-Hg refluxed 1 hr., filtered, acidified, and the ppt. filtered off, washed with H2O, dried, twice crystd. from 70% EtOH yielded 155 mg. 3-phenyl-2-hydrindenecarboxylic acid, m. 162-3.degree... V (0.2 g.) in 5 ml. 2N aq. NaOH and 0.12 g. p-ONC6H4NMe2, boiled 1 min., cooled, acidified, the brown ppt. filtered off, decompd. with 10 ml. 2N HCl, and the ppt. recrystd. from dil. alc. and AcOH yielded 96 mg. 3-phenylindenone-2-carboxylic acid, m. 155.5-6.5.degree.. HC.tplbond.COEt (7 g.) in 25 ml. abs. Et20 treated with 0.1 mole I in 60 ml. Et20, cooled in ice after the evolution of C2H6 was complete, 20 g. p-MeC6H4CHO in 50 ml. abs. Et20 added during 15 min., the soln. hydrolyzed with 30 g. NH4Cl in 150 ml. H2O, the Et2O layer washed with H2O, dried with Na2SO4, evapd. in vacuo under N, the oily residue immediately cyclized by dissolving it in 200 ml. PhMe and refluxing 2 hrs. with 1 g. p-MeC6H4SO3H, the resulting soln. washed with aq. NaHCO3, dried with Na2SO4, evapd. in vacuo, and the residue crystd. from petr. ether and alc. yielded 8 g. Et 3-p-tolyl-5-methyl-2-indene-carboxylate, m. 97-8.5.degree.. I (0.1 mole) in 150 ml. Et20 at 0.degree. treated with stirring with 0.1 mole p-O2NC6H4CHO in 125 ml. C6H6, hydrolyzed with 30 g. NH4Cl in 150 ml. H2O, worked up as usual, the residue, which crystd. on addn. of alc. and cooling, dissolved in AcOEt, and the soln. evapd. slowly yielded 7.2 g. RCH(OH)C(:CHR)CO2Et (R = p-O2-NC6H4) (VI), yellow crystals, m. 150-2.degree. Evapn. in vacuo in the cold of the Et2O soln. originally obtained gave 41% solid, C18H17BrN2O7, m. 75.5-77.degree., which did not decomp. vigorously and could be recrystd. from alc., and on heating in a vacuum desiccator lost HBr to form VI. I (0.23 mole) in 300 ml. Et20 at O.degree. treated with 20 g. AcH in 100 ml. abs. Et2O during 45 min., the soln. hydrolyzed with 60 g. NH4Cl in 300 ml. H2O, the Et2O layer sepd., washed with H2O, dried over Na2SO4, and the Et2O evapd. in vacuo under N gave a brown oily residue which decompd. vigorously on exposure to air; dissolving it in Et20, drying over Na2SO4, and distg. yielded 15.1 g. MeCH:C(CO2Et)-CHMeOCHBrMe, b12 102-4.degree., nD20 1.4794. subsequent expt. the explosive decompn. of the initial reaction product was moderated by addn. of 5 ml. AcOH after hydrolysis with NH4Cl soln., the mixt. then washed with NaHCO3 soln., dried over Na2SO4, the Et2O evapd. in vacuo, the residual brown oil (40 g.) immediately refluxed 5 min. with 100 ml. 2N H2SO4, the mixt. extd. with Et2O, the ext. washed with NaHCO3 soln., dried over Na2SO4 and the residue fractionated in vacuo, yielding 15 g. MeCH:C(CHMeOH)CO2Et (VII), b15 103-6.degree., nD20 1.4532. VII (5 g.) in 20 ml. Me2CO oxidized with 4 g. CrO3 in 10 ml. H2O and 3 ml. H2SO4 at 0-5.degree. during 2.5 hrs., 500 ml. H2O added, the mixt. extd. 3 times with Et2O, the ext. washed with aq. NaHCO3 and H2O, dried over Na2SO4, the solvent evapd., and the residue distd. in vacuo yielded 3 g. MeCH: CAcCO2Et (VIII), b14 97-9.degree., nD20 1.4513. In a similar expt. with I and EtCHO, the product immediately refluxed with 2N H2SO4 and worked up as usual yielded 33% impure EtCH:C(CO2Et)CH(OH)-Et (IX), b20 124-28.degree., nD20 1.4566. IX with CrO3 in Me2CO yielded impure EtCH:C(CO2Et)COEt (X), b15 114-16.degree., nD20 1.4520. MeCH(OH)C.tplbond.COEt (from AcH and LiC..tplbond.:COEt) (7 g.) in 60 ml. Et2O added with stirring to 0.08 mole EtMgBr in 60 ml. Et2O evolved C2H4, 6.4 g. BzH and 40 ml. Et2O then added, the mixt. worked up as with II and the residue distd. after vigorous decompn. took place, yielded 6.6 g. brown oil, b10 125-45.degree., which was sapond. by boiling with 40 ml. 10% alc. KOH, the mixt. evapd. in vacuo, the residue dissolved in H2O, extd. with Et2O, and acidified, yielded a yellow oil which crystd. on

stirring with petr. ether; recrystn. from H2O with C gave 2 g. CH2: CHC(CO2H):CHPh, m. 88-9.degree.. The dibromide, prepd. in CCl4 and crystd. from C6H6, m. 170-1.degree..

L9 ANSWER 53 OF 70 CAPLUS COPYRIGHT 2003 ACS on STN

AN 1956:77477 CAPLUS

DN 50:77477

OREF 50:14520h-i,14521a-f

TI Action of mixed organomagnesium compounds on diketene

AU Gibaud, Alain; Willemart, Antoine

SO Bulletin de la Societe Chimique de France (1956) 432-41 CODEN: BSCFAS; ISSN: 0037-8968

DT Journal

LA Unavailable

The action of PhMgBr on diketene (I) gives AcPh (II) and Ph2MeCOH (III) by AΒ cleavage (A) and dehydroacetic acid (IV) by condensation (B), both reactions occurring simultaneously. Alkylmagnesium compds. give very small yields by cleavage and anhyd. MgI2 in situ gives quant. yields of IV by condensation. Investigation of the mechanism of reactions A and B failed to give unequivocal choice between the acetylketene, cyclobutanedione, 2-buteno-.beta.-lactone, and 3-buteno-.beta.-lactone structures proposed for I. PhMgBr (0.3 mole) in 300 cc. Et20 treated slowly with vigorous stirring with 0.1 mole pure I in 200 cc. anhyd. Et20, hydrolyzed by gradual addn. of 150 cc. semisatd. NH4Cl and 0.5N HCl for several hrs., decanted, and the washed Et2O layer concd. to 75 cc., dried, treated with 25 cc. petr. ether, and evapd. slowly yielded 6.5 g. III [dehydrated to Ph2C:CH2, b12.5 136.degree., nD15.5 1.6094; PhCHBrCH2Br, m. 80.degree. (decompn.)]. Distn. of the mother liquors in vacuo gave 1.6 II, some Ph2, and a viscous fraction, yielding 1.4 g. III when crystd. from Et2O-petr. ether. Distn. of these mother liquors gave more II and III, bringing the total yields to 25% II and 40% III. The aq. layer acidified, extd. with Et2O, concd., chilled, and the product recrystd. from H2O yielded 20% IV, m. 108.degree.. Similar reactions with I were carried out with various organomagnesium compds. (organomagnesium compd., % yields of II, III, IV): MeMgI, <2, <2, 30; EtMgBr, <2, <5, 30; BuMgBr,</pre> 8, 15, 25; C6H11MgBr, 10, 12, 20; PhMgBr, 25, 40, 20; PhMgI, 5, 10, 20; PhLi, 25, 40, 20; Ph2Mg, 30, 50, 0; and 1-C10H7MgBr, 40, 0, 0. A mixt. of 0.05 mole I and 0.15 mole PhMqBr in 150 cc. tetrahydrofuran treated with 0.15 mole BzCl, refluxed 2 hrs., 120 cc. tetrahydrofuran distd. and the residue shaken violently with 250 cc. Et20, 13.2 cc. dioxane added, and the ppt. filtered off over celite, washed with Et2O, and distd. in vacuo yielded 25 g. yellow liquid, Cl(CH2)40Bz, b14 162-70%, and 3.5 g. red residue, which, mineralized with H2SO4, hydrolyzed with 15% HCl in Et2O, and recrystd. from alc. gave Bz2CH2 (V), m. 76.degree.. The Et2O-insol. ppt. taken up in H2O, extd. with a large vol. of Et2O, distd., the Et2O distd., and the residue washed with alc. and crystd. from 125 cc. C6H6 gave 0.5 g. Bz3CH (VI), m. 245.degree.. The action of BzCl on the complexes formed by the action of PhMgBr on I, producing V and VI, can be explained by the assumption of any of the 4 proposed constitutional formulas for I. The isolation of a compd. with a Mg content corresponding to the formula BzCH: CPhOMgBr is not regarded as sufficient proof for the AcCH:CO formulation. MeMgI (0.2 mole) in 200 cc. Et2O treated with 0.05 mole I in 200 cc. Et2O, hydrolyzed, extd. several times with Et2O, and the ext. distd. gave 8 cc. of a mixt. of iso- and tert-BuOH. Powd. Mg (0.26 g.) and 2.67 g. iodine in 25 cc. anhyd. Et20 treated dropwise with 1.6 cc. pure I, and the mixt. hydrolyzed with 10 cc. H2O, decolorized with Na2S2O3, decanted, washed, dried, and evapd. yielded 1.58 g. IV, m. 106.degree.. Detn. of active H by the Zerevitinov method with a Roth

microapp. for 100 moles I gave with RMgX (X = Cl, Br, and I, resp.) (R and % active H given): Me, -, 35, 43; Et, 33, 34, 42; iso-Pr, 36, 33, -. I itself has no active H; the liberated gases are generated by the HX liberated during the polymerization of the I-MgI2 complex. Similar results were obtained with LiAlH4. The lack of an active H in I contradicts the assumption of prototropic equil. forms of I proposed by Wasserman (C.A. 43, 1320d).

L9 ANSWER 54 OF 70 CAPLUS COPYRIGHT 2003 ACS on STN

AN 1956:73983 CAPLUS

DN 50:73983

OREF 50:13902i,13903a-d

TI Heterocyclic compounds. XXXV. Condensation of tetrahydro-.gamma.-pyrones and tetrahydro-.gamma.-thiapyrones with organomagnesium and lithium compounds

AU Nazarov, I. N.; Golovin, E. T.

CS Inst. Org. Chem., Acad. Sci., U.S.S.R., Moscow

SO Zhurnal Obshchei Khimii (1956), 26, 477-83 CODEN: ZOKHA4; ISSN: 0044-460X

DT Journal

LA Unavailable

- cf. C.A. 47, 5371c; 50, 9411h; preceding abstr. To PhMgBr from 9.5 g. AΒ PhBr there was added with cooling 7 g. 2,5-dimethyltetrahydro-4-thiapyrone (I) in Et2O and after 1 hr. at room temp. and 15 hrs. at reflux, the mixture was treated with ice and dil. HCl, yielding 37% 2,5-dimethyl-4-phenyltetrahydrothiapyran-4-ol (II), b2 123-4.degree.; the yield was 41% after 20 hrs. refluxing. PhLi from 1 g. Li and 12 g. PhBr in Et20 under N was treated at -10.degree. with 7 g. I; after standing overnight, the mixt. was refluxed 3 hrs. and treated with H2O, yielding 65% II, b1.7 122-3.degree.. This oxidized with KMnO4 in Me2CO in the presence of 10% H2SO4 to II sulfoxide, m. 231-2.degree. (from EtOH), relatively insol. in C6H6. Use of a larger amount of KMnO4 gave the corresponding sulfone, m. 163-4.degree. (from C6H6-petr. ether), and its diastereoisomer, m. 170-70.5.degree. (mixed m.p. 159-68.degree.). PhMgBr with 2,2-dimethyltetrahydro-4-thiapyrone gave in 2 hrs. of refluxing 68% 2,2-dimethyl-4-phenyltetrahydrothiapyran-4-ol, b2 119-20.degree., which on standing deposited a low yield of the solid form, m. 69-70.degree.. This with KMnO4 in Me2CO in the presence of 10% H2SO4 gave the sulfone, m. 180-1.degree. (from MeOH), the same being formed from the liquid form. PhMgBr with 2,2-dimethyltetrahydro-4-pyrone after 2 hrs. at room temp. gave 76% 2,2-dimethyl-4-phenyltetrahydropyran-4-ol, isolated as the liquid isomer, b2.5 104-5.degree., and solid isomer, m. 122.5-3.degree.; the former solidified and m. 122-3.degree.. 2,2-Dimethyltetrahydro-4thiapyrone and MeMgI gave in 2 hrs. 60% 2,2,4-trimethyltetrahydrothiapyran-4-ol, m. 82-3.degree. (from petr. ether). The use of 2,2dimethyltetrahydro-4-pyrone similarly gave 64% 2,2,4-trimethyltetrahydro-4pyranol, bl 56-8.degree., nD20 1.4569.
- L9 ANSWER 55 OF 70 CAPLUS COPYRIGHT 2003 ACS on STN
- AN 1956:44544 CAPLUS
- DN 50:44544

OREF 50:8610b-i,8611a-f

- TI Action of Grignard reagents. VIII. Action of organo-magnesium and lithium compounds on benzo-, naphtho-(2',3')oxazol-2-ones and their N-substituted derivatives
- AU Mustafa, Ahmed; Asker, Wafia; Hishmat, Orkede Hassan
- CS Cairo Univ., Egypt
- SO Journal of the American Chemical Society (1955), 77, 5127-30

CODEN: JACSAT; ISSN: 0002-7863

DT Journal

AB

LA Unavailable

cf. C.A. 50, 2601h. Treatment with Grignard reagents, followed by hydrolysis, caused the opening of the hetero ring in benzo- and in naphtho(2',3')oxazol-2-ones and their N-aroyl- and N-arylsulfonyl derivs. to give the corresponding N-aroyl- and N-arylsulfonyl derivs. of o-H2NC6H4OH (I) and 3,2-H2NC10H6OH (II). Similar results were obtained with PhLi. Similarly, the action of PhMgBr yielded 3-oxo-1-phenyl -1-hydroxyisoindoline (III) and Ph3COH (IV) from N-benzoylphthalimide (V), benzotriazole (VI) and IV from 1-benzoylbenzotriazole (VII), and (CH2CPh2OH)2 (VIII) and p-MeC6H4SO2NH2 in the case of N-(ptoluenesulfonyl) succinimide (IX). PhMgBr also brought out the cleavage of the N-C bond in PhSO2NPhBz (X) to give PhSO2NHPh and IV. Benzoxazol-2-one (XI) (1 q.) in 30 cc. dry C6H6 added to PhMgBr from 0.9 g. Mg and 9 g. PhBr in 50 cc. dry Et20, the Et20 evapd., the residual mixt. heated 3 hrs. on the steam bath, held at room temp. overnight, poured slowly into 100 cc. satd. aq. NH4Cl, and extd. with Et2O, the Et2O-C6H6 soln. dried and evapd., and the solid residue recrystd. from C6H6 gave 0.58 g. N-Bz deriv. (XII) of I, m. 164.degree.; it gave a greenish brown color with FeCl3 soln. Naphtho(2',3')oxazol-2-one (XIII) in 40 cc. C6H6 added to PhMgBr gave about 0.72 g. N-Bz deriv. (XIV) of II, colorless crystals, m. 233.degree. (from EtOH). 3-Benzoylbenzoxazol-2-one (XV) in 30 cc. C6H6 added portionwise to PhMgBr, the mixt. refluxed 4 hrs., held at room temp. overnight, decompd., and extd. with Et2O, the ext. evapd., and the solid residue washed with petr. ether and extd. with hot ligroine gave 0.51 g. colorless crystals of XII; the petr. ether ext. evapd. gave 0.28 g. IV, $\ensuremath{\text{m}}.$ 163.degree.. XV in 50 cc. dry C6H6 treated slowly with PhMgBr under a pos. N pressure of 7 mm., and the mixt. refluxed 3 hrs. and worked up in the usual manner gave 0.46 g. XII and 0.25 g. IV. p-MeC6H4MgI (from 0.8 g. Mg and 7.5 g. p-MeC6H4I in 40 cc. Et2O) treated with XV in 40 cc. C6H6, the mixt. worked.up as usual, the Et20-C6H6 soln. washed with about 45 cc. 10% aq. NaOH, dried, and evapd., the oily residue scratched and cooled, the resulting mixt. of colorless crystals and oil washed with about 15 cc. petr. ether to leave (p-MeC6H4)2, and the petr. ether soln. concd. and cooled gave 0.16 g. (p-MeC6H4)2C(OH)Ph, m. 75.degree.; the alk. washings acidified and extd. with Et2O, the ext. dried and evapd., and the solid residue recrystd. from C6H6 qave about 0.44 q. o-(p-MeC6H4CO)NHC6H4OH, colorless crystals, m. 135.degree.; it gave a green color with H2SO4 and a yellow color with alc. FeCl3. 1-C10H7MgBr (from 1.2 g. Mg, 10.4 g. 1-C10H7Br, and 40 cc. dry Et2O) refluxed 3 hrs. with 1.5 g. XV in 40 cc. C6H6 yielded in the usual manner 0.58 g. (1-C10H7)2C(OH)Ph (XVI), colorless crystals, m. 165.degree. (from glacial AcOH); it gave a deep violet color with H2SO4; the alkali ext. from the processing acidified with cold dil. HCl and extd. with Et20, and the ext. evapd. gave 0.87 g. o-(1-C10H7CO)NHC6H4OH (XVII), colorless crystals, m. 192.degree. (from C6H6); it gave with H2SO4 a green color which changed to brown on the addn. of a crystal of KNO3. XVII (0.5 g.) treated with 1 cc. BzCl in the presence of 10 cc. 15% aq. NaOH gave 0.61 g. O-Bz deriv. of XVII, colorless crystals, m. 174.degree. (from glacial AcOH). PhLi (from 1.2 g. Li and 12 g. PhBr), in 38 cc. Et20 added dropwise to 1.2 g. XV in 35 cc. C6H6, the mixt. refluxed 0.5 hr., held 1 hr. at room temp. (all under 7 mm. pos. N pressure), poured slowly into 100 cc. satd. aq. NH4Cl, and extd. with Et20, the ext. washed with 40 cc. 8% aq. NaOH and H2O, dried, and evapd., and the residue recrystd. from ligroine yielded 0.47 g. IV, m. 163.degree.; the alkali washings acidified gave 0.72 g. XII. XIII treated with BzCl gave almost 100% 3-Bz deriv. (XVIII) of XIII, colorless crystals, m. 226.degree.; it gave a pale yellow color with H2SO4. XVIII

in 40 cc. C6H6 treated in the usual manner with PhMgBr, the Et2O-C6H6 soln. evapd., the residue extd. with about 25 cc. cold C6H6, and the insol. residue recrystd. from EtOH gave 0.42 g. XIV, colorless crystals, m. 232.degree.; the C6H6 ext. concd. and cooled yielded 0.39 g. IV. XVIII (1.5 g.) treated similarly with 1-ClOH7MgBr gave 0.51 g. XVI and 0.88 g. IV. 3-Acetylbenzoxazol-2-one (1.5 g.) in 40 cc. C6H6 gave similarly with PhMgBr 0.83 g. XII and 0.58 g. Ph2C(OH)Me, m. 82-3.degree. (it gave a red color with H2SO4). XI (1 g.) in 10 cc. freshly distd. pyridine treated with 1.5 g. PhSO2Cl, the mixt. heated 0.5 hr. on the steam bath, kept at room temp. overnight, and filtered, and the residue washed with about 10 cc. cold EtOH and recrystd. from hot EtOH gave 1.32 g. 3-benzenesulfonylbenzoxazol-2-one (XIX), colorless crystals, m. 144.degree.. XIX (1.5 g.) and PhMgBr worked up in the usual manner, the Et20 soln. washed with 40 cc. cold aq. NaOH and H2O, dried, and evapd. gave 0.46 g. IV; the alkali washings acidified and extd. with Et2O gave 0.78 q. N-Bz deriv. (XX) of I, m. 140-1.degree.; it gave an olive-green color with alc. FeCl3. XX (0.5 q.) treated with 1 cc. PhSO2Cl in the presence of 20 cc. 15% aq. NaOH, the mixt. heated 2 hrs. on the steam bath, and the resulting colorless solid recrystd. from EtOH gave about 64% O, N-di-Bz deriv. of I, m. 164.degree.. 3-(p-Toluene-sulfonyl)benzoxazol-2one and PhMgBr gave similarly 0.46 g. IV and 0.58 g. o-(p-MeC6H4SO2NH)C6-H4OH (XXI), which gave a pale green color with alc. FeCl3 and yielded, treated with BzCl and aq. NaOH, the O-Bz deriv. 3-PhSO2 deriv. (XXII) of XIII, colorless crystals, m. 204.degree., was prepd. in almost 100% yield from XII and PhSO2Cl in the usual manner. XXII (0.7 g.) treated with PhMgBr in the usual manner gave 0.18 g. IV and 0.27 g. 3,2-PhSO2NHC10H6OH, m. 166.degree. (from C6H6); it gave an olive-green color with alc. FeCl3. XIX treated with 1-C10H7MgBr, the mixt. worked up in the usual manner, the Et20 soln. washed with aq. NaOH and H2O, dried, and evapd., the oily residue treated with 20 cc. fuming HNO3, the mixt. poured into cold H2O, and the yellow powdery ppt. washed with AcOH gave hexa-nitro-.alpha.,.alpha.,.alpha.-trinaphthylcarbinol; the alk. washings acidified gave 0.38 g. XX. XIX (1.5 g.) treated with PhMgBr gave 0.54 g. IV and 0.51 g. XX. V in 50 cc. C6H6 treated with PhMgBr in the usual manner, the Et20 soln. evapd., the oily residue washed with hot petr. ether, and the resulting powder recrystd. from C6H6-petr. ether yielded 0.38 g. III, m. 163.degree.; it gave a red color with H2SO4; the petr. ether washings gave 0.21 q. IV. VII and PhMqBr gave similarly 0.62 g. IV and VI, m. 98.degree., which treated with BzCl yielded VII. IX treated with PhMgBr and the mixt. worked up in the usual manner yielded 0.62 g. VIII, colorless crystals, m. 205-6.degree., and from the alk. exts. 0.32 g. p-MeC6H4SO2NH2, colorless crystals. X and 30 cc. C6H6 added to PhMgBr, the mixt. refluxed 5 hrs., held at room temp. overnight, decompd. with aq. NH4Cl, and extd. with Et2O, the ext. washed with 35 cc. 10% ag. NaOH and H2O, and evapd., and the oily residue washed with cold petr. ether and crystd. from CHCl3 gave 0.53 g. IV; the alk. washings acidified and extd. with Et20 gave 0.21 g. PhSO2NHPh.

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L9 ANSWER 56 OF 70 CAPLUS COPYRIGHT 2003 ACS on STN
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AN 1956:44254 CAPLUS

DN 50:44254

OREF 50:8446c-h

TI The deuterium isotope effect in the methanolysis of some organometallic compounds

AU Wiberg, Kenneth B.

CS Univ. of Washington, Seattle

SO Journal of the American Chemical Society (1955), 77, 5987-90 CODEN: JACSAT; ISSN: 0002-7863

- DT Journal
- LA Unavailable
- The Bu, Ph, and PhCH2 Grignard and Li reagents react with MeOD faster or AΒ as fast as with ordinary MeOH. A similar isotope effect was noted in the reaction of MeCHNaCO2Me (I) with MeOH. The results are compared with the data available in the literature. The isotope effect of the neutralization of the C-anion of PhCH(OH)CN (II) also was detd., and the rate law for the benzoin condensation has been reconsidered based on this evidence. MeOH and D2O fractionated through a column gave MeOD. BuMgBr (0.1 mole) in 80 cc. Bu2O refluxed in a slow stream of N, the mixt. treated with 1 cc. MeOD in 20 cc. Bu2O and heated, and the resulting C4H10 swept with N into a Dry Ice-Me2CO trap gave C4H10 contg. 33.5 .+-. 0.2% D. BuMgBr (from 2.1 g. BuBr and 0.28 g. Mg) in 15 cc. Bu20, refluxed in a slow stream of N and then added slowly to 5 cc. MeOD contg. 33.5-4.5% D in 15 cc. Bu20 with stirring, and the resulting butane isolated in the usual manner gave in 3 identical runs butane with 38.8, 38.9, and 36.3% D (isotope effect kH/kD 0.83, 0.83, and 0.87), resp. A similar reaction with an equiv. amt. Li for the Mg gave quite erratic results and low yields of butane which were overcome by using C6H6 as the solvent. BuBr gave thus with Li and MeOD contg. 33.5% D in 2 runs butane contg. 33.6 and 33.7% D (isotope effect 1.00 and 0.99), resp. PhMgBr from 3.1 g. PhBr, 0.50 Mg, and 15 cc. dry Et20 added with stirring to 8 cc. MeOD contg. 33.5 and 36.7% D, the mixt. treated after 10 min. with dil. HCl, and the org. layer dried and distd. gave C6H6, b. 78-82.degree., contg. 34.4 and 37.5% D, resp. (isotope effect 0.96, 0.97); the C6H6 contg. small amts. of Et2O and Bu20 which did not interfere with the mass spectrum analysis. PhLi and MeOH contg. 34.5 and 33.5% D gave C6H6 contg. 37.1 and 35.7% D (isotope effect 0.89 and 0.91), resp. PhCH2MgCl from 2.5 g. PhCH2Cl and 0.50 g. Mg added to MeOD contg. 33.5 (36.7)% D, and the mixt. worked up in the usual manner gave toluene contg. 35.0 (36.9)% D [isotope ratio 0.93 (0.99)]. EtCO2Me (3 cc.) added with shaking to 100 cc. 0.45N Ph3CNa, the resulting Et2O soln. of I added with stirring to 15 cc. MeOD contg. 75.7% D, the mixt. treated after 0.5 min. with stirring rapidly with 55 cc. N HCl, and the Et2O soln. washed, dried, and distd. gave 1.5 cc. EtCO2Me, b. 77-9.degree., contg. 72.8% D (isotope effect 1.16). The benzoin condensation carried out as described previously (C.A. 49, 13184e) but with 93.7% pure EtOD showed after 75 and 120 min. 23 and 32% exchange with rate consts. ke of 3.5 and 3.2 .times. 10-3, resp.; with 52.0% pure EtOD 10 and 16% exchange occurred after 75 and 120 min., resp., with an av. rate const. ke of 1.5 .+-. 0.1 .times. 10-3.
- L9 ANSWER 57 OF 70 CAPLUS COPYRIGHT 2003 ACS on STN
- AN 1956:35956 CAPLUS
- DN 50:35956
- OREF 50:7072d-g
- TI Addition reactions of triazenes, illustrating the reactivity of N:N double bonds
- AU Klages, Friedrich; Mesch, Walter
- CS Univ. Munich, Germany
- SO Chemische Berichte (1955), 88, 388-96 CODEN: CHBEAM; ISSN: 0009-2940
- DT Journal
- LA Unavailable
- AB When azo compds. are converted to triazenes, the N:N double bonds become less ready to undergo nucleophilic addn. reactions so that triazene derivs. cannot be obtained by such reactions (cf. Gilman and Pickens, C.A. 19, 2936). Reaction of PhN:NNHPh (I) with EtMgBr in Et2O or 'tetrahydrofuran leads to elimination of C2H6 and recovery of I when the

red complex is hydrolyzed with dil. aq. NH4Cl. I is also recovered when the red complex of I with PhLi in Et2O or C6H6 solns. is destroyed, or on decompn. of the complex of I with LiAlH4, m. 245.degree., in Et2O or tetrahydrofuran. Reaction of I with EtMgBr in Et20 and refluxing with BzCl leads to formation of N; on hydrolysis PhNBz2 (II), m. 161.degree., is formed, while the same reaction at 0.degree. gives PhN2Cl (III) as well as II. Reaction of EtMgBr in Et20 with PhN:NHMe, then with BzCl, gives C2H6, N2, and II. PhN:NNBzPh with BzCl in Et2O in the presence of SbCl5, BF3, ZnCl2, or MgBr2 with cooling yields III after hydrolysis. EtMgBr with PhN:NNMe2 (IV) in tetrahydrofuran gives C2H6, N, and, after hydrolysis of the complex, 1,4-diphenyl-2,5-diethylhexahydro-1,2,4,5tetrazine, m. 124.degree., as well as MeNH2. An addn. mechanism is proposed for its formation, which is accompanied by a secondary reaction in which the N is eliminated. IV with dry LiAlH4 at 120.degree. gives PhNH2 and NHMe2, this reaction can be regarded as reduction with decompn. of the N chain. p-MeC6H4N:NNMe2 (from PhNMe2 and p-MeC6H4N2X), m. 51.degree., with PhLi in Et2O forms N and m-MeC6H4CH2NHMe, characterized as the H oxalate, m. 205.degree., and as the p-toluenesulfonamide, m. 81.degree.; a mechanism is presented for this reaction.

L9 ANSWER 58 OF 70 CAPLUS COPYRIGHT 2003 ACS on STN

AN 1956:16132 CAPLUS

DN 50:16132 OREF 50:3300c-g

TI Reactions of the carbonamide group. III. Reaction with organometallic compounds

AU Heyns, Kurt; Pyrus, Wolfgang

CS Univ. Hamburg, Germany

SO Chemische Berichte (1955), 88, 678-83 CODEN: CHBEAM; ISSN: 0009-2940

DT Journal

LA Unavailable

AB cf. C.A. 48, 1261c. Amides, R'CONHR'' (including polypeptides), react with a 3- to 5-fold excess of RMgX or RLi, in suitable solvents and at sufficiently high temp., to produce, after hydrolysis, R'COR + H2NR''. AcNHMe, PhNHAc (I), Et hippurate, BzNHCH2CPh2OH (II), Et aceturate, Et N-glycylglycinate (III), H2NCH2CONHCH2CPh2OH (IV), or Me N-(N-leucylglycyl)glycinate (V) reacted thus. R in the RLi was Ph (at 36.degree.) or Et; reaction times for RLi were 1-3 hrs. R in RMqX was Ph (at 150.degree. in most cases), Me, Et, or Bu; times were 4-30 hrs. Yields of R'COR were only about 20-40%, and the reaction is therefore not practical for preparative cleavage of proteins. Caprolactam and BuMgBr at 150.degree. gave 2-butyl-.DELTA.1-hexamethylenimine 14.5% and 2,2-dibutylhexamethylenimine 6.7%, isolated as the picrolonates, m. 173.degree. and 118.degree., resp. H in -CONH- reacts with MeMgI in the Zerevitinov detn. if the detn. is performed in anisole at 120.degree.; compds. tested and moles of CH4 evolved were: I, 1.02 and 1.00; II, 2.01 and 2.02; AcNHCH2CPh2OH (VI), 2.01 and 1.99; III.HCl, 3.82 and 3.84; V.HCl, 4.78 and 4.88; IV, 4.71 and 4.78; N-qlycylqlycine, 0.02 and 0.01; 2,4-piperazinedione, 0.01 and 0.03. EtMgBr and I, heated 1 hr., then treated with AcCl or PrCOCl, gave PhNAc2 or PhNAcCOPr, but II or VI, treated in the same way, were dehydrated to 53% BzNHCH: CPh2, m. 131.degree., or 46% AcNHCH:CPh2 (VII), m. 161.degree., resp. VII was prepd. also from VI and Ac2O at 150.degree. in a sealed tube. VI, m. 138.degree., was prepd. from Et aceturate and PhMgBr. .alpha.-Aminoisocaprophenone-HCl, m. 199-203.degree. (decompn.) (2,4-dinitrophenylhydrazone-HCl, m. 202.degree.), was prepd. by reaction of concd. HCl at 135.degree. in a sealed tube with N-(1-benzoy1-3-

methylbutyl)benzamide (VIII), m. 107.degree.. VIII was obtained from N-benzoylleucyl chloride and AlCl3 in C6H6. .alpha.-Aminoacetophenone 2,4-dinitrophenylhydrazone-HCl m. 221.degree..

- L9 ANSWER 59 OF 70 CAPLUS COPYRIGHT 2003 ACS on STN
- AN 1955:73515 CAPLUS
- DN 49:73515
- OREF 49:13953h-i,13954a-i,13955a-f
- Action of Grignard reagents. VI. (a) Cleavage by organomagnesium and lithium compounds and by lithium aluminum hydride; (b) action of phenyllithium on phenanthraquinone and benzil monoximes
- AU Mustafa, Ahmed; Asker, Wafia; Hishmat, Orkede H.; Shalaby, Ahmed F. A.; Kamel, Mohamed
- CS Cairo Univ., Egypt
- SO Journal of the American Chemical Society (1954), 76, 5447-52 CODEN: JACSAT; ISSN: 0002-7863
- DT Journal
- LA Unavailable
- cf. C.A. 48, 12084b. The treatment with Grignard reagents, followed by AB hydrolysis caused opening of the hetero ring in substituted 1,8-naphthosultones to give the corresponding derivs. of 8-arylsulfonyl-1-naphthol. PhMgBr also caused the cleavage of the N-S bond in N-arylsulfonyl derivs. and the cleavage of the N-C bond in N-aroyl derivs. Similar results were obtained with PhLi. The hydrogenolysis with LiAlH4 caused the opening of the hetero ring of 1,8-naphthosultone (I) and its substituted derivs. and of N-phenylsulfonylnaphthosultam (II) to give the corresponding disulfides. 5-Br deriv. (III) of I (1 g.) in 50 cc. dry Et20 added to PhMgBr from 0.9 g. Mg and 9 g. PhBr in 50 cc. dry Et20, the mixt. heated 3 hrs. on the steam bath, kept overnight at 25.degree., poured slowly into 100 cc. satd. aq. NH4Cl, dried, filtered, and evapd., the oily residue washed several times with hot petr. ether, and the resulting solid recrystd. from C6H6 gave 0.73 g. 5,8-Br(PhSO2)C10H5OH (IV), m. 162.degree., brown in H2SO4, gave a violet color with alc. FeCl3. III (1.5 g.) and 1-C10H7MgBr gave 0.9 g. 5.8-Br(1-C10H7SO2)C10H5OH (V), m. 195.degree., green in H2SO4, gave a red-brown color with alc. FeCl3. The 5-BrCH2 deriv. (VI) (2.0 g.) of I gave similarly 1.1 g. 5-BrCH2 analog (VI) of IV, m. 169.degree., purple in H2SO4, gave a red color with alc. FeCl3; and 1.2 q. 5-BrCH2 analog (VII) of V, m. 232.degree., blue-green in H2SO4, gave a red color with alc. FeCl3; all products were sol. in aq. NaOH with yellow color. VI (1 g.) in 40 cc. Et2O treated with CH2N2 [from 10 g. H2NCON(NO)Me] in Et2O 24 hrs. at 0.degree., the mixt. washed with about 40 cc. 8% cold aq. NaOH and H2O, dried, and evapd., and the residue recrystd. from C6H6 gave 78% Me ether of VI, m. 199.degree.. VII gave similarly 71% Me ether of VII, m. 244.degree.. A mixt. of PhMgBr and (PhSO2)2NPh (VIII) worked up in the usual manner, the oily residue washed with petr. ether and then extd. with hot petr. ether, and the insol. product dissolved in C6H6 and pptd. with petr. ether gave 0.19 g. Ph2SO2, m. 124.degree.; the pert. ether ext. concd. and cooled gave 0.41 g. PhSO2NHPh; the light petr. washings slowly evapd. gave 0.13 g. Ph2, 1-(p-MeC6H4SO2)2NC10H7 (IX) gave similarly 0.42 g. p-(1-C10H7NHSO2)C6H4Me and 0.23 g. p-MeC6H4SO2Ph. N-p-Toluenesulfonylcarbazole (X) and PhMgBr gave similarly a solid residue which washed several times with ligroine (b. 60-80.degree.) and recrystd. from EtOH gave 0.51 g. carbazole, m. 240.degree.; the concd. ligroine washings gave 0.1 g. p-MeC6H4SO2Ph. PhNBz2 (XI) and PhMgBr refluxed 5 hrs., the mixt. kept at room temp. overnight, decompd. with dil. HCl, and extd. with Et2Q, and the insol. product washed with H2O and recrystd. from EtOH gave 0.6 g. BzNHPh, m. 162.degree.; the Et2O-C6H6 soln. washed with about 45 cc. 8% aq. NaOH,

dried, and evapd. yielded 0.27 g. Ph3COH, m. 163.degree. (from C6H6-petr. ether). 1-C10H7NBz2 (XII) and PhMqBr gave in the usual manner 0.53 g. 1-C10H7NHBz and 0.23 g. Ph3COH. N-Benzoylcarbazole (XIII) gave similarly 0.41 g. carbazole and 0.27 g. Ph3COH. BzNHPh (2 g.) in 75 cc. C6H6 and PhMgBr refluxed 8 hrs. and decompd. with dil. HCl gave 0.38 g. BzNHPh, and from the C6H6-Et2O mixt. 0.61 g. Ph2C: NPh. 1-C10H7NHBz and PhMgBr gave about 0.7 g. recovered starting material and 0.33 g. 1-C10H7N:CPh2, yellow, m. 136.degree.. MgI (from 1 g. Mg powder and 4 g. iodine in 20 cc. dry Et20 and 20 cc. dry C6H6) refluxed 3 hrs. with N,N'-dibenzenesulfonyldianthranilide (XIV), kept at room temp. overnight, and decompd. with ice in satd. aq. NH4Cl gave essentially unchanged XIV; VIII and X behaved in the same manner. PhLi from 16 g. PhBr, 1.5 g. Li. and 100 cc. dry Et2O added to 2 g. XIV in 50 cc. dry C6H6, the mixt. kept at room temp. overnight under N at a pos. pressure of 7 mm., poured slowly into 100 cc. satd. aq. NH4Cl, extd. with Et2O, the ext. dried and evapd., and the oily residue washed several times with cold petr. ether and recrystd. from C6H6-petr. ether gave 1.7 g. .omicron.-PhSO2NHC6H4C(OH)Ph2, m. 188.degree.. 2-Phenyl-1,2-benzisothiazol-3-one 1,1-dioxide treated with PhLi (from 1 q. Li and 12 q. PhBr in 80 cc. Et20) in 30 cc. C6H6, the mixt. decompd. with dil. HCl, the org. layer evapd., and the oily residue washed with light petroleum and crystd. from C6H6 gave 0.58 q. .omicron.-PhNHSO2C6H4C(OH)Ph2, m. 205.degree.. 2-Phenylsulfonyl-1,2benzisothiazol-3-one in 30 cc. C6H6 treated with PhLi in Et2O, the mixt. extd. with about 27 cc. cold 8% aq. NaOH, and H2O, dried, and evapd., and the oily residue washed twice with 60 cc. petr. ether (b. 40.degree.) and crystd. from petr. ether gave 0.41 g. .omicron.-PhSC6H4C(OH)Ph2, m. 140.degree.; the alk. ext. acidified with dil. HCl, extd. with Et20, dried, and evapd., and the solid residue (0.29 g.) crystd. from C6H6 gave PhSO2NH2. N-Benzenesulfonylphthalimide (2 g.) in 50 cc. C6H6 treated in the usual manner with PhMgBr, the Et2O layer extd. with cold aq. NaOH, washed with H2O, dried, and evapd., and the oily residue washed with petr. ether and recrystd. gave 1.1 g. .omicron.-C6H4[C(OH)Ph2]2, m. 201.degree.; the petr. ether washings slowly evapd. gave 0.31 g. Ph2; the aq. alk. ext. acidified with dil. HCl gave 0.48 g. PhSO2NH2. I (2 g.) treated with PhLi and 30 cc. C6H6, the mixt. kept 0.5 hr. at 25.degree. and decompd., the Et20-C6H6 layer washed with about 60 cc. 8% cold aq. NaOH, the alk. ext. acidified, extd. with Et20, dried, and evapd., and the residue recrystd. from C6H6-petr. ether gave 8,1-PhSO2C10H6OH, m. 140.degree., gave a green color with alc. FeCl3. VIII, IX, X, XI, XII, and XIII gave with PhLi instead of PhMqBr the same reaction products with no marked difference in VIII (1 q.) in 30 cc. C6H6 added in portions to 0.7 q. LiAlH4 in 50 cc. Et2O previously refluxed 15 min., the mixt. refluxed 3 hrs., kept at room temp. overnight, treated with cold dil. HCl, dried, and evapd., and the oily residue washed several times with petr. ether and recrystd. gave 0.34 g. PhSO2NH2; the light petr. washings evapd. gave an oil which treated with BzCl and ag. NaOH gave 0.22 g. BzSPh. XIII and XII gave similarly 0.42 g. carbazole and 0.38 g. BzNHPh; the petr. ether washings gave PhCH2OH. I, VI, the CH2Cl, and the Me deriv. refluxed 2 hrs. with LiAlH4 in the usual manner, the mixt. treated with cold aq. NH4Cl, the org. layer dried and evapd., the oily residue from I washed with petr. ether, and the washings evapd. gave 0.1 g. 1-hydroxy-8-thionaphthol, m. 58.degree., gave a blue-green color with alc. FeCl3; the residue from the washings recrystd. from C6H6-petr. ether gave 0.45 g. 1,1'-dihydroxy-8,8'dinaphthyl disulfide (XV), yellow, m. 168-9.degree., brown in H2SO4; the solid residue from the CH2Cl and Me derivs. recrystd. from C6H6-petr. ether gave 65, 72, and 78%, resp., of the 4,4'-dimethyl deriv. (XVI) of XV, pale yellow crystals, m. 128.degree., olive-green in H2SO4. BzCl (2 cc.) added to 0.4 g. XV in 20 cc. 8% aq. NaOH, and the resulting solid

washed with H2O, dried, and recrystd. from C6H6 gave almost 100% di-Bz deriv. of XV, m. 189-90.degree., yellow-green in H2SO4 changing to brown. Similarly was prepd. in almost quant. yield the di-Bz deriv. of XVI, m. 260.degree.. II (1 g.) treated with LiAlH4 in the usual manner yielded 0.52 g. 1,1'-di(phenylsulfonylamino)-8,8'-dinaphthyl disulfide, colorless crystals, m. 179.degree., yellow in H2SO4 changing to olive-green (red-brown in the presence of a crystal of KNO3). Phenanthraquinone monoxime (1 g.) in 40 cc. dry C6H6 treated with PhLi, the C6H6 soln, dried and evapd., and the solid residue recrystd. from C6H6 gave 71% 10-phenanthrylhydroxylamino-9-hydroxyphenanthrene, m. 162.degree.. A-Benzil monoxime gave similarly 1,1,2-triphenyl-1-oximino-2-ethanol, m. 153-4.degree., in almost quant. yield.

L9 ANSWER 60 OF 70 CAPLUS COPYRIGHT 2003 ACS on STN

AN 1955:73466 CAPLUS

DN 49:73466

OREF 49:13929d-i,13930a

TI The reaction of Grignard reagents with .alpha.,.beta.-unsaturated sulfones. I

AU Potter, Howard

CS Alma Colli, Alma, MI

SO Journal of the American Chemical Society (1954), 76, 5472-4 CODEN: JACSAT; ISSN: 0002-7863

DT Journal

LA Unavailable

The behavior of .alpha.,.beta.-unsatd. sulfones is variable according to AΒ the degree of substitution at the .beta.-C, being similar to the behavior of comparable unsatd. ketones in yielding products of conjugate addn. or of alternative reactions, but different in that the reaction with PhLi is influenced the same as is the reaction with PhMgBr. BzCH2Cl (172 g.) in MeOH and 1 equiv. of p-MeC6H4SNa refluxed, the mixt. concd. to a small vol., the residue shaken with H2O and Et2O, the Et2O layer evapd., and the residue distd. gave 0.42 mole PhAc and 168 g. p-MeC6H2SCH2Bz (I), b5 182-4.degree., m. 46.degree., which was oxidized with H2O2 in 100% yield to p-MeC6H4SO2CH2Bz, m. 107.degree.. Approx. 0.5 mole PhMgBr added with stirring and cooling to 100 g. I, the mixt. kept 10 min. at 0.degree., and shaken with iced HCl, and the Et2O layer extd. with aq. NaOH (gave acidified 24 g. p-MeC6H4SH), steam distd., dried, and distd. gave 32 g. p-MeC6H4SPh (II), b5 131-2.degree.; at 200-20.degree. bath temp. and 3-5 mm. p-MeC6H4SCH:CPh2 (III), m. 84.degree. (from Me2CO-petr. ether or glacial AcOH-MeOH); at 280-300.degree. p-MeC6H4SCH2C(OH)Ph2, oil, was obtained. III treated 2 hrs. at 10.degree. with 1 equiv. 30% H2O2 in glacial AcOH the mixt. poured into H2O and extd. with Et2O, and the ext. washed, dried, and evapd. gave 50% p-MeC6H4SOCH:CPh2 (IV), m. 124-5.degree. (pptd. from Me2CO with petr. ether); the crystn. residues oxidized gave p-MeC6H4SO2CH:CH2 (V). III treated with 2 equivs. H2O2 or IV treated with 1 equiv. H2O2 gave 100% V, m. 103-3.5.degree. (from MeOH). V was oxidized with KMnO4 in Me2CO to PhBz. V added to 5 mole equivs. PhMgBr, the mixt. let stand a day or longer, (or C6H6 added, the Et2O $\,$ distd. off, and the C6H6 soln. refluxed), treated with ice and acid, the org. layer washed and steam distd., the steam distillate extd. with Et2O, the ext. dried and evapd., the residue treated with 30% H2O2 in glacial AcOH, the AcOH soln. dild. with H2O and steam distd., and the distn. residue recrystd. from MeOH gave p-MeC6H4-SO2Me (VI), m. 123-5.degree., the same result was obtained with IV. III and IV gave similarly with PhLi impure VI from which some Ph2SO2, m. 114-17.degree., was isolated. The residue from the 1st steam distn. treated with H2O2 in glacial AcOH and recrystd. from MeOH gave V, m. 103.degree.. p-MeC6H4-SO2CH:CHPh gave with

PhLi similarly as PhMgBr 85% p-MeC6H4CO2CH2CHPh2, m. 150-1.degree. (from MeOH). I (16.7 g.) in hot C6H6 treated with vigorous stirring dropwise with 0.05 mole PhMgBr vaporizing the Et2O through a column, the mixt. refluxed 1 hr., kept 24 hrs. at room temp., and worked up in the usual manner gave only 14.5 g. unchanged I. PhMgBr (0.25 mole) treated with 0.05 mole I in C6H6, the mixt. let stand overnight, the Et2O distd. off, the C6H6 soln. refluxed 3 hrs., and the mixt. worked up in the usual manner by steam distn. gave 3.5 g. white gleaming crystals, m. 58-60.degree. (from MeOH), contg. 94.5% C and 6.2% H; this hydrocarbon could not be found among the products after 6 days at room temp. and was not investigated further.

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L9 ANSWER 61 OF 70 CAPLUS COPYRIGHT 2003 ACS on STN
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AN 1954:23530 CAPLUS

DN 48:23530

OREF 48:4234f-i

TI Sulfonates

IN Helberger, Johann H.; Heyden, Rudi W. F.

PA Bohme Fettchemie G. m. b. H.

DT Patent

LA Unavailable

FAN.CNT 1

PATENT NO. KIND DATE APPLICATION NO. DATE

PI DE 895598 19531105 DE

A process of prepq. sulfonates, useful as detergents, wetting, textile AΒ auxiliary, or pharmaceutical agents, or intermediates in the manuf. of dyes, comprises treating sultones with hydrocarbon compds. of metals of Group I, II, or III of the periodic table, such as Grignard reagents, BuLi, PhNa, and fluorene Na. A mixt. of a soln. of Na 1.15 in EtOH 25, fluorene (I) 8.4, and decahydronaphthalene 100 parts by wt. is heated until no more EtOH distills off. The sultone of hydroxybutanesulfonic acid (II) (7 parts) is added, and the reaction is achieved by heating at 150-80.degree.. The solvent is removed by distn., and the residue is extd. with ether to remove unreacted I. A readily water-sol. product Na 4-(9-fluorenyl)-2-butanesulfonate 10 parts of good wetting and detergent properties is thus obtained. The sultone of hydroxypentanesulfonic acid may be used in place of II. A product of the probable formula C6H13SO3.MgBr is similarly prepd. from II and EtMgBr, a product of the probable formula C6H5C4H8SO3MqBr from II and PhMqBr, and a colorless hygroscopic powder from PhLi and II. Cf. preceding abstr.

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L9 ANSWER 62 OF 70 CAPLUS COPYRIGHT 2003 ACS on STN
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AN 1954:14291 CAPLUS

DN 48:14291

OREF 48:2575c-g

TI Application of ultrasonic waves to the preparation of organometallic compounds

AU Renaud, Pierre

SO Bulletin de la Societe Chimique de France (1950) 1044-5 CODEN: BSCFAS; ISSN: 0037-8968

DT Journal

LA Unavailable

AB The effect of ultrasonic waves on the prepn. of organometallic compds. of Li, Ca, Hg, Al, Be, and Zn is studied. They do not overcome the inertia of halogens in the formation of Grignard reagents. The prepn. of organo compds. of Li under the influence of ultrasonic waves takes place rapidly in Et2O even at 66.degree. B.acte.e. but is impossible in Bu2O. Derivs.

of Ca cannot be obtained even by aid of EtMgI. Hg emulsifies immediately in Et20 but does not react with bromides. Al affords organo-Al compds. by reaction of an organo-Mg deriv. on Al powder; the use of Al-Mg alloy is unnecessary. Organo-Zn compds are not obtained by an analogous reaction between RMgX and Zn in the presence of Cu. Beryllium does not resemble Al in its action. The halides of Fe, Ni, and Co give condensation reactions, catalyzed by a complex such as [CoCl2(NH3)4]Cl but not by [CoCl3(NH3)3]. In the production of Grignard compds. chlorides do not react, even with excitement by a bromide, except when Cl is mobile as in PhCH2Cl. CHCl3 and CCl4 retain their inhibiting action. Generally, only aliphatic or aromatic iodides and bromides react within a few min. or more slowly if a solvent (Et20, Bu20) is used. EtBr and PhBr do not attack Mg in the absence of a solvent. MeCH:CHBr does not furnish an organo-Mg deriv. even in presence of HgCl2. HC.tplbond.CHCH2Br only gives such a deriv. in the presence of HgCl2; the reaction is more rapid than that induced by simple heating. Mg 2-pyridyl bromide is obtained without heating but the further reaction with AcH is impossible without recourse to heat. EtMqBr, BuMqBr, and PhMqBr are obtained with the aid of ultrasonic waves in slightly ag. Et20 or Bu20, contq. up to 50% of C6H6, light petroleum, or even palm oil.

. L9 ANSWER 63 OF 70 CAPLUS COPYRIGHT 2003 ACS on STN

AN 1953:58583 CAPLUS

DN 47:58583

OREF 47:9936a-i,9937a-h

TI The reaction of organometallic compounds on quinol. I

AU Wessely, F.; Holzer, L.; Vilcsek, H.

CS Univ. Vienna

SO Monatshefte fuer Chemie (1952), 83, 1253-73 CODEN: MOCMB7; ISSN: 0026-9247

DT Journal

LA Unavailable

cf. Wessely and Sinwel, C.A. 45, 7545e. Reaction of RMgX (or PhLi) with AB an o-quinol acetate (I) unsubstituted meta to the keto group takes place by a 1,4-addn. to the C:C.C:O system, followed by splitting out of H2O, to give a phenol with R (or Ph) in the m-position. With a p-quinol acetate (II) having a free o-or m-position to the keto group, reaction takes place by 1,2-addn., with splitting out of H2O and migration of R or Ph; 1,4-addn. is not observed. o-PhC6H4OH (III) (6 g.) with 1.5 mole Pb(OAc)4 (cf. C.A. 45, 1972b) in 800 ml. HOAc, kept 2 hrs. at room temp., evapd., the residue treated with H2O, extd. with ether, the ext. washed with NaHCO3, dried, and concd., and the ppt. filtered washed with cold ether gave 3.8 g. crude 2-phenyl-o-quinol acetate (IV) (2.5 g. from dil. alc.), m. 128-30.degree.. IV with Pd-C and H gave III. p-PhC6H4OH (V) gave 4-phenyl-p-quinol acetate (VI), m. 106-9.degree. (from ether-petr. ether), and mesidine gave 2,4,6-trimethyl-p-quinol acetate (VII), m. 56-7.degree. (from ether-petr. ether). RMgX, made in the usual way to give about a 2.5M soln. in ether, filtered from the excess Mq, added dropwise to I or II (about 0.3M soln. in ether) (to give a molar ratio of RMgX to I or II of about 4:1), the soln. heated 5-10 min. on a water bath, cooled, treated with satd. NH4Cl, the layers sepd., the ether soln. extd. with N NaOH, and the ext. acidified and extd. with ether gave the acid fraction (A); the ether soln. gave the neutral fraction (N). The following products were thus obtained from A and/or N: With 0.64 g. 2-methyl-o-quinol acetate (VIII) and MeMgI: A, 0.15 g. o-cresol (IX) (aryloxyacetic acid, m. 152-3.degree.) and 0.05 g. 2,5-Me2C6H3OH (aryloxyacetic acid, m. 114-15.degree.). With 2 g. VIII and EtMgBr: A, a little IX and 0.9 g. 2,5-MeEtC6H3OH (aryloxyacetic acid, m. 99-100.degree.). With 1 g. VIII and iso-BuMgBr; only IX. With 3 g.

VIII and PhMgBr: A, 1.7 g. 2,5-MePhC6H3OH(XI) (an addnl. 0.6 g. XI was obtained from N by extn. with 10% NaOH), b0.02 140-5.degree., m. 70.degree. (m. 78.degree. after long standing alone or in soln.; this melt when cooled gave the form m. 70.degree.) (the Me ether was prepd.). With 1 g. IV and MeMgI: A, 0.6 g. phenol, distd. twice at 100-10.degree./0.2 mm., to give 2,5-PhMeC6H3OH (XII) (no cryst. aryloxyacetic acid), whose constitution was verified by paper chromatography, N, 0.3 g. brown oil. With 1.74 g. 2,4,6-trimethyl-o-quinol acetate (XIII) and MeMgI: A, only mesitol (XIV); N, 0.5 g. light terpenelike oil. With 0.9 g. XIII and EtMgBr: A (and also N) 0.6 g. 2,4,6,3-Me3EtC6HOH (XV), b0.01 60-80.degree., m. 96-7.degree. (from petr. ether) (Me ether, b12 105-10.degree.). With 5 g. XIII and PhMgBr: A, only XIV; N, extd. with 10% NaOH, 1 g. 2,4,6,3-Me3PhC6HOH (?) (XVI) (C analysis, 0.65% low), b0.08 120-5.degree. [took up only 1.8 equivs. AcO with Pb(OAc)4, was not nitrated with fuming HNO3 in Ac20-HOAc (1:1) at -10.degree., did not react with 3,5-(O2N)2C6H3COCl and ClCH2CO2H, and gave BzOH with alk. KMnO4], Ph2, Ph2C(OH)Me (XVII), and C15H16O, m. 113.degree., insol. in boiling N With 4-methyl-p-quinol acetate (XVIII) and MeMgI: only 2,4-Me2C6H3OH (XIX). With 1.8 g. XVIII and PhMgBr: A, p-cresol, b0.1 75-80.degree., and 0.8 g. 2,4-PhMeC6H3OH (XX); N, Ph2, b0.1 80-100.degree., and from the residue after distn. of Ph2, by extn. with 10% NaOH, an addnl. 0.5 g. XX, m. 67-8.degree. (from petr. ether) (3,5-dinitro-benzoate, m. 115-16.degree.). XX, converted with Me2SO4 to the Me ether and oxidized with the calcd. amt. of 0.5N KMnO4 in Na2CO3, gave 4,3-MeOPhC6H3CO2H (XXI), b0.005 150-60.degree.. With 0.68 g. VI and MeMgI: A, 0.5 g. mixt., not sepd. by distn. at 120.degree./0.005 mm., sepd. by crystn. from ether-petr. ether to give V, m. 157-60.degree., and $0.\bar{2}$ g. $\bar{2}$, 4-MePhC6H3OH (XXII), m. 112-13.degree. (many times recrystd. from petr. ether); N, distd. at 100-20.degree./0.005 mm., XXII. With 5 g. VII and EtMgBr: A, 0.2 g. XIV; N, 4.1 g. terpenelike oil. To 2.2 moles Li free of oxide, cut in small pieces, suspended in about half the abs. ether needed to make a 0.5M soln. of PhLi, cooled, stirred under dry, H-free N, was added (30 min.) 1 mole PhBr in the other half of the ether, the mixt. stirred 20 min. more, filtered, and the soln. used as reagent. I or II (0.1M) in abs. ether treated dropwise the PhLi soln. with stirring under O-free N (1 mole I or II to 5 moles PhLi), ice-cold concd. NH4Cl soln. added, the layers sepd., the aq. soln. extd. 1-2 times with ether, the ether solns. extd. with N or 10% NaOH, the alk. soln. acidified and extd. with ether gave A; the ether soln. gave N. With PhLi and 1.48 g. VIII: A, 1 g. brown oil which on distn. gave a small amt. of IX, b0.008 50.degree., and a fraction, b0.008 110-20.degree., which was taken up in C6H6, filtered (to give dark red needles, m. 108-12.degree., whose compn. was not studied), put on an Al2O3 column, and eluted with ether to give XI, m. 72.degree.; N, 2.5 g. brown oil which was distd. gave PhAc, b0.005 70.degree., and a 2nd fraction, b0.005 95-105.degree., which on redistn. gave a mixt. of XI and XVII (sepd. by removal of XI with 10% NaOH). When 1 mole PhLi and 1 mole VIII were used, two-thirds of VIII was recovered, no biphenol was isolated, and small amts. of PhAc and IX were formed. With PhLi and 1.72 g. XIII: A, a small amt. of brown oil; N, 2.5 g. somewhat cryst. substance, distd. to give PhAc, b0.1 95-110.degree., and · 1.7 g. colorless liquid, b0.1 120-30.degree., giving on redistn. 1.3 g. main fraction, b0.008 95-105.degree., which was shaken with 80 ml. 10% NaOH, the resulting solid filtered, washed well with H2O, and sublimed (80-90.degree./0.008 mm.), giving 0.35 g. XVII, m. 79-80.degree.; the alk. soln. acidified, extd. with ether, the ext. dried, concd., and the residue distd., gave XVI, b0.008 105-10.degree. (with the same properties as XVI obtained from XIII and PhMgBr). With PhLi and 3.46 g. XVIII: A, XXII and a small amt. of unknown brown oil, b0.025 110-20.degree.; N; PhAc, XVII,

and XXII. 4,3-Me-(O2N) C6H3NH2.H2SO4 (10 g.) in 12 ml. cold HCl (1:1) diazotized with 3.5 g. NaNO2 in 10 ml. H2O, treated with 5 g. MgSO4 and 40 ml. C6H6, 35 ml. 15% NaOH added dropwise, the mixt. dild. with C6H6, the layers sepd., the C6H6 soln. dried, concd., the residue steam-distd., the distillate extd. with ether, the ext. concd., and the residue dissolved in alc. gave on cooling in Dry Ice 4,3-Me(O2N)C6H3Ph (XXIV), m. 61-2.degree.. XXIV (84 mg.) with H and Raney Ni gave 68 mg. 3-amino compd. (XXV), b0.01 110.degree., m. 54-5.degree.. XXV, diazotized and heated in 20% H2SO4, gave XI, b0.005 115.degree., m. 72.degree. (from petr. ether), after several days, m. 78.degree.. Acetomesitylene (6 g.) with 15 g. Zn-Hg and 60 ml. HCl (1:1) with C6H6 and some HOAc, refluxed 6 days, and the product heated with Na at 150.degree. and distd., gave 2,1,3,5-EtC6H2Me3 (XXVI), b18 95-6.degree., nD19 1.5071. Picramide diazotized with NaNO2 in 100% H2SO4 at 35-40.degree., XXVI in HOAc added dropwise, the resulting azo compd., m. 168.degree. (decompn.)(crude), heated with SnCl2 and concd. HCl, the mixt. made basic, and the amine extd. with ether, purified by extn. in 2N HCl, distd. (b0.05 60.degree.), and treated with HCl gas in abs. ether, gave the HCl salt, m. 180.degree. (decompn.), which, diazotized in HCl (1:2), refluxed, and steam-distd., gave XV, b12 80-90.degree., m. 95-7.degree.. Ph2 nitrated to a mixt. of o- and p-nitro compds., the p-isomer, m. 114.degree., sepd. from the o-isomer, m. 35-6.degree. (which was purified by chromatography, the 1st fraction being eluted with alc.). The p-isomer with Raney Ni and H gave 100% p-PhC6H4NH2 (XXVII), m. 48-9.degree.. To 15 q. XXVII.HCl in 90 ml. HOAc and 90 ml. abs. dioxane, stirred and cooled in an ice-salt bath, was added (1 hr.) excess EtONO, the resulting diazo salt treated at 0.degree. with concd. aq. urea until N evolution ceased, warmed on a water bath as more N evolved slowly, the mixt. heated 2 hrs. on a steam bath, cooled, extd. with ether, the phenol purified by extn. with 10% NaOH, pptn. with acid, and reextn. with ether, and the ext. washed, dried, and concd., giving 6-7 g. V, m. 162-4.degree. (from petr. ether). V (5 g.) refluxed 3 hrs. with 6.25 g. CHCl3, 6.25 g. NaOH, and 156 ml. H2O, the mixt. filtered hot (the filter cake gave unchanged V), and the filtrate acidified, gave crude 3,6-Ph(HO)C6H3CHO (XXVIII), m. 85-95.degree., purified by 2 chromatographs on acid-Al2O3, eluted with C6H6 (XXVIII was in the 1st eluate) and recrystn. from ether-petr. ether, m. 99-100.degree.. XXVIII (0.2 g.) refluxed 5 hrs. with 1.5 ml. alc., 0.6 ml. HOAc, 6 ml. HCl(1:2), and 0.2 g. Zn-Hq, dild. to twice the vol. with H2O, extd. 4 times with ether, the ext. dried and concd., and the residue sublimed at 110-20.degree./0.005 mm., gave XXII, m. 113-14.degree.. 3,4-Ph(MeO)C6H3Ac (Slotta and Nold, C.A. 30, 2944.4) and Br in NaOH soln. (Johnson, et al., C.A. 40, 7172.1) gave 95% XXI, m. 219-21.degree.. Rf values (in C6H6) are given for III 0.84, XII 0.85, XX 0.85, V 0.49, XXII 0.66, and XI 0.66. Ultraviolet absorption curves are given for XVI, XI, XXX, XIV and its Me ether, and XV and its Me ether.

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L9 ANSWER 64 OF 70 CAPLUS COPYRIGHT 2003 ACS on STN
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DN 46:11360

OREF 46:2009f-i

TI The reaction of trimethylene oxide with Grignard reagents and organolithium compounds

AU Searles, Scott

CS Northwestern Univ., Evanston, IL

SO Journal of the American Chemical Society (1951), 73, 124-5 CODEN: JACSAT; ISSN: 0002-7863

DT Journal

LA Unavailable

AN 1952:11360 CAPLUS

- Dropwise addn. of 60 g. Cl(CH2)3OAc during 45 min. to 65 g. NaOH, 65 g. AΒ KOH, and 5 g. H2O at 150-60.degree. and purification (cf. Noller, C.A. 44, 2443i) gave 42-5% (CH2)30 (I). General procedure: addn. of 0.13-0.20 mole I in 3 vols. anhyd. Et2O to 0.18-0.30 mole RMgX (or RLi occasionally) in cold Et20 (formation of a white ppt.), refluxing 1 hr., addn. of 150-200 cc. C6H6, removal of the Et2O by distn., refluxing 4 hrs., cooling, hydrolysis with satd. NH4Cl, extn. with Et2O or CCl4, and distn. of the org. solns. gave the desired R(CH2)30H (II), characterized generally as the 3,5-dinitrobenzoate or the 1-naphthylurethan. Data (read R and % yield II): Ph, 84% (also 4% Br(CH2)3OH) (III); Ph, 85% from PhLi; 1-ClOH7, 80%; 2-C10H7, 60%; 9-fluorenyl, 44% from RLi; PhCH2, 83%; Bu, 28% from BuLi; cyclohexyl, 28% II, also 40% III; Me2CH, 28% II and 12% III; Me3C, 37% Cl(CH2)3OH. Data for new II (read b.p. and nD20): 1-Cl0H7, bl 118-19.degree., 1.615 (phenylurethan, m. 75-6.degree.); 2-C10H7, b7 120-1.degree. (phenylurethan, m. 94.degree.); 9-fluorenyl, b0.2 141.degree. (1-naphthylurethan, m. 124-5.degree.). III was prepd. in 54% yield from 0.2 mole I and 0.5 mole anhyd. MgBr2.
- L9 ANSWER 65 OF 70 CAPLUS COPYRIGHT 2003 ACS on STN
- AN 1948:713 CAPLUS
- DN 42:713
- OREF 42:147h-i,148a
- TI Mechanism of addition of Grignard reagents to nitriles
- AU Swain, C. Gardner
- CS Massachusetts Inst. Technol., Cambridge
- SO Journal of the American Chemical Society (1947), 69, 2306-9 CODEN: JACSAT; ISSN: 0002-7863
- DT Journal
- LA Unavailable
- AB The reaction of BuMgBr and PhCN in ether was followed by measuring the evolution of gas from aliquot samples taken periodically from the reacting soln. It is homogeneous and 2nd order with a rate const. of 3.7 .+-. 0.6 .times. 10-4 l. mole-1 sec.-1 at 25.degree. and 5.8 .+-. 0.9 .times. 10-5 at 0.degree. This corresponds to a half-life of 7.6 hrs. with 0.1 M reactants at 25.degree. and to an activation energy of 12 .+-. 1 kcal. The observed kinetic order is consistent with a suggested mechanism involving as its rate-detg. step the intramol. rearrangement of a complex, present in a low concn. in rapid equil. with the organometallic reagent and addend. PhLi and PhMgBr show at least a 100-fold difference in reactivity with PhCN.
- L9 ANSWER 66 OF 70 CAPLUS COPYRIGHT 2003 ACS on STN
- AN 1946:27676 CAPLUS
- DN 40:27676
- OREF 40:5418e-i,5419a-d
- TI Method of reaction of metallo-organic compounds. VI. The reaction between organometallic compounds and ethers or thio ethers
- AU Luttringhaus, Arthur; Saaf, Grete Wagner-v.; Sucker, Elfriede; Borth, Gunther
- SO Ann. (1945), 557, 46-69
- DT Journal
- LA Unavailable
- PhoCH2CH:CH2 (9 g.), added to Ph2Mg (prepd. by concg. 150 cc. of a 0.38 M soln. until the b.p. is 75.degree.) and boiled 6 hrs., gives 4.9 g. PhOH and 5.3 g. of CH2:CHCH2Ph. Octanol (26 g.) and 4.8 g. Na in 60 cc. PhMe, boiled until soln. is complete and then boiled 8 hrs. with 35 g. CH2:CHCH2Br, give octyl allyl ether (I), bl2 87-8.degree.; I (17 g.) and 0.12 M PhMgBr, heated 6 hrs. at 75.degree., give 85% of CH2:CHCH2Ph and

70.5% of octanol (isolated as the benzoate). BuSCH2CH:CH2 (II) (10 g.) and 1.8 equivs. of PhMgBr, heated 9 hrs. at 74.degree., give 9 g. of unchanged II; 2 equivs. of BuMgBr, heated 9 hrs. at 80-6.degree., gives 91% of unchanged II. PhSCH2CH: CH2 (III) (15 g.) and 0.13 mole PhMgBr, heated 6 hrs. at 78.degree., give 48% PhCH2CH: CH2 and 6 g. of III, together with 35% of PhSH. p-CH2:CHCH2OC6H4Ac (17.6 g.) in 20 cc. ether, added dropwise to PhMgBr (from 40 g. PhBr, 6 g. Mg, and 90 cc. ether) and boiled 4 hrs., gives 8 g. PhCH2CH: CH2 and 13.5 g. (70%) of p-hydroxy-1,1-diphenylethylene, b0.03 114-16.degree., m. 56.degree. (benzoate, m. 79.degree.); Me2SO4 gives the known Me ether, m. 75.degree.. m-BrC6H4OCH2CH:CH2 (2.13 g.) with 10 g. activated Mg in 30 cc. ether, refluxed 8 hrs. and the product decompd. with dil. AcOH, gives 4.2 g. of m-CH2:CHCH2C6H4OH, bl3 108-15.degree. (characterized as the Me ether, bl2 90-3.degree., d2018 0.992, which is oxidized to m-MeOC6H4CO2H). PhOCH2Ph (IV) is relatively stable toward Grignard reagents. With PhLi in ether (2 hrs. at 20.degree., after which the soln. is added to solid CO2), 11 q. of IV yields 7 g. unchanged IV, 1.5 g. Ph2CHCH2Ph, 0.8 g. BzOMe, and 0.6 g. Ph2CHCO2Me (prepd. from the intermediate Ph2C(CO2H)2); after 5 hrs. at 20.degree. the products include 2.5 g. Ph2CHCH2Ph, 0.6 g. BzOMe, and 1.4 q. Ph2CHCO2Me. No Ph2CHCH2Ph is formed at 0.degree. in 0.5 hr. Ph2CH2 (0.1 mole) and 75 cc. N PhLi in ether, allowed to stand 5 hrs. at 20.degree. and the soln. decompd. with CO2, give 4.8 g. Ph2C(CO2Me)2, m. 93-4.degree.; after heating with PhLi for 6 hrs. at 55.degree., 60% of Ph2CH2 is unreaeted. A preparative method for Ph2C(CO2H)2 consists in heating Ph2CH2 with 2 mols. of PhLi for 6 hrs. at 80.degree., followed by decompn. with CO2. PhOBu and PhNa in ether, shaken 24 hrs. at room temp. and warmed 6 hrs. at 55.degree., give only 47% PhOH. Ph dodecyl ether (V) (39.3 g.) and PhNa (from 0.25 mole PhCl), shaken 20 hrs. at room temp. and warmed 8 hrs. at 60.degree., give 78% of 2-dodecene (VI), b13 96-7.5.degree., some Ph2, 8.5 g. PhOH, and 0.9 g. of a fraction b0.9 180-210.degree. (not studied). VI (9 g.), treated at 3.degree. with BzO2H and the oxide boiled 6 days with 20 cc. dioxane and 30 cc. H2O, gives 3.5 g. of 2,3-dihydroxydecane (VII), m. 68-9.degree.; with Pb(OAc)4 VII yields AcH; the mother liquor from VII yields a mixt. of HCHO and AcH; thus VI consists of approx. 40% of 1- (VIII) and 60% of 2-dodecene. If the Na complex from V is decompd. with CO2, and the fraction b0.25 125-30.degree. reduced over Raney Ni at 25.degree., there results C12H25CO2H, m. 41 degree. Synthetic VIII (17 g.) and iso-AmNa (from 55 g. iso-AmCl and 30 g. Na) in 100 cc. cyclohexane, shaken 20 hrs. at room temp. and 8 hrs. at 60.degree., and the product decompd. with CO2, give 5.5 g. of unchanged VIII and 13.8 g. of an acid fraction which, after reaction with CH2N2 yields 4.8 g. of a mixt. of Me tridecenoates; hydrogenation and fractionation yield 1.6 g. of C12H25CO2H and 1.9 g. of Me 1-ethylhendecanoate, b0.03 95-6.degree., whose amide m. 105.degree. (the synthesis from EtCK(CO2Et)2 and C9H19Br is reported). Ph2S (23 q.) and PhNa (from 17 g. PhCl) in 80 cc. C6H6, shaken 10 hrs. at room temp. and heated 30 hrs. at 70.degree., give 3.3 g. PhSH, 4.8 g. Ph2S, 7.7 g. (C6H4)2S, and 2.8 g. of a resin. Ph2S (5.2 g.) and 50 cc. 0.5 N PhLi in Pr20, heated 170 hrs. at 80.degree., give 4.1 g. Ph2S and 0.59 g. (C6H4)2S. Ph2O and Ph3CNa give CPh4. (C6H4)2O and (C6H4)2S are not affected by long heating with PhNa at 70-80 degree. The mechanism of these reactions is discussed.

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L9 ANSWER 67 OF 70 CAPLUS COPYRIGHT 2003 ACS on STN
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AN 1943:19064 CAPLUS

DN 37:19064

OREF 37:3079c-e

TI Factors determining the course and mechanism of Grignard

reactions. IX. The effect of metallic halides on the reaction of organolithium compounds with organic halides

- AU Kharasch, M. S.; Lewis, Daniel W.; Reynolds, W. B.
- SO Journal of the American Chemical Society (1943), 65, 498-500 CODEN: JACSAT; ISSN: 0002-7863
- DT Journal
- LA Unavailable
- Details are given of the reaction of PhBr with PhLi, BuLi and BuMgBr in the absence of CoCl2 and in the presence of 5 mole-% of CoCl2 and also of BuBr on PhLi and Bu-MgBr under the same conditions. The coupling reaction, which predominates in the uncatalyzed reaction, is almost completely suppressed. The products from PhLi and BuBr may be readily accounted for by a chain mechanism in which a cobalt subhalide acts as the chain carrier. The differences between the final products in the expts. with Li and Mg are readily accounted for by the metathetical equil. which has been observed in the case of the Li compds. However, this does not explain the formation of octane in the Li expts. and its absence in the Mg expts.
- L9 ANSWER 68 OF 70 CAPLUS COPYRIGHT 2003 ACS on STN
- AN 1941:47679 CAPLUS
- DN 35:47679
- OREF 35:7368a-f
- TI Factors determining the course and mechanisms of **Grignard**reactions. IV. The effect of metallic halides on the reaction of al Grignard reagents and organic halides
- AU Kharasch, M. S.; Fields, E. K.
- SO Journal of the American Chemical Society (1941), 63, 2316-20 CODEN: JACSAT; ISSN: 0002-7863
- DT Journal
- LA Unavailable
- AB. Co does not react with PhBr at 40.degree. after 1 hr. PhMgBr (I) gives 6-8% of Ph2 (II). I (0.14 mole) and 9 mol.% of CoCl2 (III) give 27% II; in the following the figure before the metal halide is mol.%; 0.54 mole I, 0.4 mole PhBr (IV) and 7 III give 83% II; 0.113 mole I, 0.1 mole IV and 2.5 III give 86% II. IV takes part in this reaction, as can be demonstrated by a halogen titration of the ag. soln. obtained by hydrolysis of the reaction mixt. after the reaction has ceased and by the fact that only a portion of IV can be recovered; thus, IV acts as an oxidizing agent in converting I into II. The II is formed exclusively from I because IV can be replaced by p-BrC6H4Me (with 9 III 86% II), EtBr (with 7 III 81% II) or iso-PrCl (with 5 III 58% II). The org. radical of IV is responsible for the formation of higher-boiling compds.; thus, 0.54 mole I, 0.4 mole II and 7 III in ether (heated 2 hrs.) give 18 g. C6H6, 34.5 g. Ph2, 1.7 g. terphenyl, 0.8 g. quaterphenyl and 17.5 g. very high-boiling material; such compds. are not found with aliphatic halides. PhCl (4 III) gives only 37% II. PhMgI and 28 III in ether-C6H6 at 0.degree. for 3 hrs. give 64% II; with 0.1 mole IV, 4 III gives 86% II. With I and IV other metal halides give the following results: 9 CuCl 6% II, 4 MnCl2 21% II, 5 FeCl3 47% II, 4 NiCl2 72% II, 4 CrCl3 7% II. Other reactions were also studied; thus, p-MeC6H4MgBr and IV with 10 III give 95% (p-MeC6H4)2; o-MeC6H4MgBr and EtBr with 7 III give 75% of (o-MeC6H4)2; p-MeOC6H4MgBr and EtBr with 5 III give 76% of (p-MeOC6H4)2; o-EtOC6H4MgBr and EtBr with 5 III give 74% of (o-EtOC6H4)2. It is believed that these reactions proceed through the agency of a Co subhalide, the active chain carrier; suggested reactions are: I + III .fwdarw. PhCoCl + MqBrCl; 2PhCoCl .fwdarw. Ph2 + 2CoCl; CoCl + PhBr .fwdarw. CoClBr + Ph-; x(Ph-) .fwdarw. C6H6, Ph2, C6H4Ph2, (6H4Ph)2, etc. These equations are used to

explain the results obtained in part III. The data point to a definite relation between the electronegativity of the org. radical, the stability of the intermediate organometallic compd. and the rate of the normal addn.

L9 ANSWER 69 OF 70 CAPLUS COPYRIGHT 2003 ACS on STN

AN 1939:41310 CAPLUS

DN 33:41310

OREF 33:5823b-f

TI The reactions of ald-chlorimines with Grignard reagents

AU LeMaistre, J. W.; Rainsford, A. E.; Hauser, Charles R.

SO Journal of Organic Chemistry (1939), 4, 106-10 CODEN: JOCEAH; ISSN: 0022-3263

DT Journal

LA Unavailable

- AΒ A study is made of the reactions taking place with ald-chlorimines (I) and Grignard reagents. Three different types of reactions can be involved, i. e., an elimination of HCl, a reaction in which the NCl group is attacked, or an addn. to the C:N bond. It was previously shown that EtONa and I in certain cases give nitriles in quant. yield. With Grignard reagents the first 2 types of reactions, with the 2nd predominating, take place. In general 0.025-0.07 mol. I prepd. according to H. and co-workers (C. A. 29, 2939.2) is dissolved in 100-200 cc. dry Et2O and the soln. in most cases cooled to 0.degree. and the RMqX is added drop by drop. After standing for a few hrs. the mixt. is filtered and hydrolyzed. An analysis of the filtrate for active Cl shows that about 10% of I remains which is converted into the aldehyde by HCl. The Et2O soln. is then allowed to stand with a satd. NaHSO3 soln. for 24-36 hrs., the aldehyde-bisulfite compd. filtered and the aldehyde liberated with Na2CO3. Evapn. of the Et20 soln. gives the nitrile. The following benzalchlorimines have been used with EtMgBr (II) or PhMgBr (III), resp., and give the following resp. % of nitriles and RCH:NMgX derivs: 2-chlorobenzal compd. and II, 13% and 43%; 4-MeO compd., 17% and 50%; the 4-Cl compd. at 0.degree. 20% and 45%, at 23-8 degree. 34% and 45%, with III 10% and 61%, with p-ClC6H4MgBr 5% and 18% in addn. to 20% PhCl and 25% p-C6H4Cl2. PhLi and 4-chlorobenzalchlorimine react to give the nitrile and a N-Li compd. which on hydrolysis yields the aldehyde. The mechanism of the various types of reactions is discussed.
- L9 ANSWER 70 OF 70 CAPLUS COPYRIGHT 2003 ACS on STN
- AN 1939:41261 CAPLUS

DN 33:41261

OREF 33:5804f-h

- TI Synthesis of primary amines by the reaction of .alpha.-methylhydroxylamine with organomagnesium and organolithium compounds
- AU Sheverdina, N. I.; Kocheshkov, K. A.
- SO Zhurnal Obshchei Khimii (1938), 8, 1825-30 CODEN: ZOKHA4; ISSN: 0044-460X
- DT Journal
- LA Unavailable
- AB RMgX and RLi in ether soln. at a temp. of -10.degree. to -15.degree. react readily with MeONH2 (I) to give primary amines. The yield depends on the nature of X, decreasing sharply from Cl to I, and is practically independent of the nature of R. The following compds. were reacted with I: EtMgBr, iso-AmMgCl, iso-AmMgBr, iso-AmMgI, sec-BuMgCl, tert-BuMgCl, PhMgCl, PhMgI, p-BrC6H4MgBr and PhLi. The yields of RNH2 were resp. 66.6, 80.1, 71.4, 5.3, 73.4, 73.6, 65.0, 0.23, 72.5 and 63.0%.

```
=> d his
```

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(FILE 'HOME' ENTERED AT 16:14:11 ON 24 SEP 2003)
     FILE 'REGISTRY' ENTERED AT 16:14:32 ON 24 SEP 2003
               STRUCTURE UPLOADED
L1
L2
            210 S L1 SSS FULL
     FILE 'CAPLUS' ENTERED AT 16:15:16 ON 24 SEP 2003
           309 S L2
L3
L4
             O S L3 AND SYN THESIS AND PREPARATION
L5
             48 S L3 AND SYNTHESIS
L6
             3 S L5 AND LITHIUM
L7
             2 S L5 AND MAGNESIUM
           194 S GRIGNARD REACTION AND LITHIUM AND MAGNESIUM
L8
L9
            70 S L8 AND PHENYL
L10
            30 S L8 AND PHENYL AND CHLORIDE
            10 S L8 AND PYRIDINE
L11
L12
             1 S L8 AND PYRIMIDINE
L13
             0 S L8 AND PYRIDAZINE
L14
             2 S L8 AND FURAN
             1 S L8 AND THIEN
L15
            84 S L8 AND CHLORIDE
L16
             O S L16 AND PHENYL AND PYRIDINE AND FURAN AND THIEN AND PYRIMIDIN
L17
             0 S L8 AND PYRIDINE AND CHLIRIDE
L18
             1 S L8 AND PYRIMIDINE AND CHLORIDE
L19
             1 S L8 AND FURAN AND CHLORIDE
L20
L21
             1 S L8 AND THIEN AND CHLORIDE
=> d l19 fib hitstr abs total
'FIB' IS NOT A VALID FORMAT FOR FILE 'CAPLUS'
The following are valid formats:
ABS ----- GI and AB
ALL ----- BIB, AB, IND, RE
APPS ----- AI, PRAI
BIB ----- AN, plus Bibliographic Data and PI table (default)
CAN ----- List of CA abstract numbers without answer numbers
CBIB ----- AN, plus Compressed Bibliographic Data
DALL ----- ALL, delimited (end of each field identified)
DMAX ----- MAX, delimited for post-processing
FAM ----- AN, PI and PRAI in table, plus Patent Family data
FBIB ----- AN, BIB, plus Patent FAM
IND ----- Indexing data
IPC ----- International Patent Classifications
MAX ----- ALL, plus Patent FAM, RE
PATS ----- PI, SO
SAM ------ CC, SX, TI, ST, IT
SCAN ----- CC, SX, TI, ST, IT (random display, no answer numbers;
             SCAN must be entered on the same line as the DISPLAY,
             e.g., D SCAN or DISPLAY SCAN)
STD ----- BIB, IPC, and NCL
IABS ----- ABS, indented with text labels
IALL ----- ALL, indented with text labels
IBIB ----- BIB, indented with text labels
IMAX ----- MAX, indented with text labels
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Patel.

ISTD ----- STD, indented with text labels

OBIB ----- AN, plus Bibliographic Data (original)

OIBIB ----- OBIB, indented with text labels

SBIB ----- BIB, no citations SIBIB ----- IBIB, no citations

HIT ----- Fields containing hit terms

HITIND ----- IC, ICA, ICI, NCL, CC and index field (ST and IT)

containing hit terms

HITRN ----- HIT RN and its text modification

HITSTR ----- HIT RN, its text modification, its CA index name, and

its structure diagram

HITSEQ ----- HIT RN, its text modification, its CA index name, its

structure diagram, plus NTE and SEQ fields

FHITSTR ---- First HIT RN, its text modification, its CA index name, and

its structure diagram

FHITSEQ ---- First HIT RN, its text modification, its CA index name, its

structure diagram, plus NTE and SEQ fields

KWIC ----- Hit term plus 20 words on either side

OCC ----- Number of occurrence of hit term and field in which it occurs

To display a particular field or fields, enter the display field codes. For a list of the display field codes, enter HELP DFIELDS at an arrow prompt (=>). Examples of formats include: TI; TI,AU; BIB,ST; TI, IND; TI, SO. You may specify the format fields in any order and the information will be displayed in the same order as the format specification.

All of the formats (except for SAM, SCAN, HIT, HITIND, HITRN, HITSTR, FHITSTR, HITSEQ, FHITSEQ, KWIC, and OCC) may be used with DISPLAY ACC to view a specified Accession Number. ENTER DISPLAY FORMAT (BIB):bib

L19 ANSWER 1 OF 1 CAPLUS COPYRIGHT 2003 ACS on STN

AN 1962:403839 CAPLUS

DN 57:3839

OREF 57:728d-i,729a-q

Reactions of .alpha.-dimethylaminophenylacetonitrile and its ethylation product with basic or nucleophilic reagents

Morris, Gene F.; Hauser, Charles R. AU

Duke Univ., Durham, NC CS

Journal of Organic Chemistry (1962), 27, 465-71 SO

CODEN: JOCEAH; ISSN: 0022-3263

DT Journal

LΑ Unavailable

=> d 120 fbib hitstr abs total

L20 ANSWER 1 OF 1 CAPLUS COPYRIGHT 2003 ACS on STN

AN1991:247788 CAPLUS

DN 114:247788

ΤI Peptide derivatives preparation as retroviral protease inhibitors

Kempf, Dale J.; Plattner, Jacob J.; Norbeck, Daniel W.; Boyd, Steven A.; Baker, William R.; Erickson, John W.; Fung, Anthony K. L.; Crowley, Steven R.

```
Abbott Laboratories, USA
PA
    PCT Int. Appl., 222 pp.
SO
     CODEN: PIXXD2
DT
     Patent
LΑ
    English
FAN.CNT 1
    PATENT NO.
                     KIND DATE
                                          APPLICATION NO.
                                                          DATE
                                          -----
PΙ
    WO 8910752
                     A1
                           19891116
                                          WO 1989-US2055
                                                            19890512
        W: AU, DK, JP, KR, US
        RW: AT, BE, CH, DE, FR, GB, IT, LU, NL, SE
                                          US 1988-194678
                                                            19880513
    EP 342541
                      A2
                            19891123
                                           EP 1989-108590
                                                            19890512
    EP 342541
                      A3
                            19911106
        R: ES, GR
                                           US 1988-194678
                                                            19880513
    AU 8935660
                            19891129
                                           AU 1989-35660
                                                            19890512
                      A1
                                           US 1988-194678
                                                            19880513
                                           WO 1989-US2055
                                                            19890512
     EP 415981
                           19910313
                                           EP 1989-905856
                                                            19890512
                      A1
        R: AT, BE, CH, DE, FR, GB, IT, LI, LU, NL, SE
                                           US 1988-194678
                                                            19880513
                                           WO 1989-US2055
                                                            19890512
     JP 03504247
                      T2
                                           JP 1989-506033
                            19910919
                                                            19890512
                                           US 1988-194678
                                                            19880513
                                           WO 1989-US2055
                                                            19890512
```

OS MARPAT 114:247788

Peptide derivs. are prepd. as retroviral protease inhibitors. Synthetic processess involved carbodiimide coupling, or coupling in combination with deprotection, and reaction with mixed anhydrides. Thus,

N-methyl-1-cyclohexenecarboxamide was treated with BuLi in THF, treated with ClTi(OPr-iso)3, and then Boc-phenylalaninal to give

N-methyl-6-[2-(tert-butoxycarbonyl)amino-1-hydroxy-3-phenyl]propyl-1-cyclohexenecarboxamide. This was then deprotected with HCl in dioxane to give N-methyl-6-(2-amino-1-hydroxy-3-phenylpropyl)-1-cyclohexenecarboxamide-HCl (I). I was coupled with Boc-Leu-Asn in the presence of 180-BuO2CCl to give the amide.

=> d 121 fbib hitstr abs total

GI

```
L21 ANSWER 1 OF 1 CAPLUS COPYRIGHT 2003 ACS on STN
     1991:114595 CAPLUS
AN
DN
     114:114595
     Novel heteroarotinoids: synthesis and biological activity
TI
     Spruce, Lyle W.; Gale, Jonathan B.; Berlin, K. Darrell; Verma, A. K.;
ΑU
     Breitman, Theodore R.; Ji, Xinhua; Van der Helm, Dick
     Dep. Chem., Oklahoma State Univ., Stillwater, OK, 74078, USA
CS
SO
     Journal of Medicinal Chemistry (1991), 34(1), 430-9
     CODEN: JMCMAR; ISSN: 0022-2623
     Journal
DT
LΑ
     English
OS
    CASREACT 114:114595
```

VII, $R=R^1=Me$, X=SVIII, R=Me, $R^1=H$, X=SIX, R=H, $R^1=Me$, X=SX, $R=R^1=H$, X=SXI, R=H, $R^1=Me$, X=OXII, $R=R^1=H$, X=O

AB Thirteen heteroarotinoids were synthesized. The key step in each prepn. was the condensation of the appropriate chroman-, thiochroman-, or benzothienyl-substituted phosphorus ylide, obtained from the independent synthesis of the corresponding phosphonium salts, with selected polyene-substituted aldehyde esters. Screening of the compds. was with one of two assays. One assay measured the ability of a retinoid to inhibit the phorbol ester induced increase of mouse epidermal ornithine decarboxylase (ODC) activity. The other assay measured retinoid-induced differentiation of the human myeloid leukemia cell line HL-60. In the ODC assay, all thirteen compds. were screened. The most active heteroarotinoids were ester I and the acid II. Both of these retinoids had ID50 values (dose required for half-maximal inhibition of phorbol ester induced ODC activity) of about 0.3 nmol. In comparison, the ID50 value for trans-retinoic acid III was 0.12 nmol while the ID50 values for acids IV and V were about 3.5 nmol. Heteroarotinoids VI and VII-XII had ID50 values of 35 nmol or greater. With a thiochroman unit, the most active acids in decreasing order of activity in the ODC assay were II > V > VI. Thus, simple replacement of the terminal propenyl system [C(16,17,18)] in IV with a cyclopropyl group produced acid VI with markedly reduced activity. With a benzoic acid group as part of the structure attached to the thiochroman unit, the ODC activity was enhanced as shown in I and II. The combination of the 2,2,4,4tetramethylthiochroman group and the benzoic acid (or ester) terminal group seemed to enhance the biol. action which resembles that found with (E) - 4 - [2 - (5, 6, 7, 8 - tetrahydro - 5, 5, 8, 8 - tetramethyl - 2 - naphthalenyl) - 1 propenyl]benzoic acid, a well-known model system. Replacing the protons with fluorine in the C(12) Me group in the side chain and altering the orientation of the aryl groups around the double bond from anti to syn lowered ODC activity in both the thiochroman- and chroman-contg. systems. Esters VII and IX and acid VIII were essentially inactive while acid X exhibited a high ID50 in the ODC assay. In the chroman family, both ester XI and acid XII had unfavorable ID50 values. Since acid VIII differs only slightly from acid X [the latter is devoid of the geminal di-Me group at C(2)] and acid X differs only slightly from acid XII, possibly the nature of the heteroatom and the stereochem. at the .alpha. position may play important roles in regulating activity, but more examples are required to

establish a trend. Changing the ring size from a fused six-six system to a five-six system led to ester Me (E)-4-[2-(2,3-dihydro-3,3-dimethylbenzo[b]thien-5-yl)-1-propenyl]benzoate (XIII) and acid (E)-4-[2-(2,3-dihydro-3,3-dimethylbenzo[b]thieny-5-yl)-1-propenyl]benzoic acid (XIV), resp. In sep. expts. from those with of I-XII and known compds both XIII and XIV exhibited similar inhibition of ODC activity to that of III at the 34 nmol level. The ID50 values of XIV and XIV were, however, 10 and 200 times greater than that of III resp. In view of the toxicity of III, ester XIII may hold promise in chemotherapy. Of eight heteroarotinoids examd. in the HL-60 assay system, only acid IV displayed modest activity. This acid had an ED50 value (dose required for half-maximal effect) of 500 nM. In comparison, the ED50 for III was 50 nM. All of the other heteroarotinoids had ED50 values which were greater than 1000 nM.

=> log y		
COST IN U.S. DOLLARS	SINCE FILE	TOTAL
	ENTRY	SESSION
FULL ESTIMATED COST	539.24	687.60
DISCOUNT AMOUNTS (FOR QUALIFYING ACCOUNTS)	SINCE FILE	TOTAL
	ENTRY	SESSION
CA SUBSCRIBER PRICE	-104.81	-104.81

STN INTERNATIONAL LOGOFF AT 16:31:38 ON 24 SEP 2003

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TERMINAL (ENTER 1, 2, 3, OR ?):2

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                 Web Page URLs for STN Seminar Schedule - N. America
NEWS
                 "Ask CAS" for self-help around the clock
NEWS
     3
         SEP 09
                 CA/CAplus records now contain indexing from 1907 to the
                 present
         Jul 15
NEWS
                Data from 1960-1976 added to RDISCLOSURE
         Jul 21
NEWS
                 Identification of STN records implemented
NEWS
         Jul 21
     6
                 Polymer class term count added to REGISTRY
     7
         Jul 22
NEWS
                 INPADOC: Basic index (/BI) enhanced; Simultaneous Left and
                 Right Truncation available
NEWS
         AUG 05
                 New pricing for EUROPATFULL and PCTFULL effective
                 August 1, 2003
NEWS 9
         AUG 13
                 Field Availability (/FA) field enhanced in BEILSTEIN
NEWS 10
         AUG 15
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NEWS 11
        AUG 15
                 PCTGEN: one FREE connect hour, per account, in
                 September 2003
NEWS 12
        AUG 15
                 RDISCLOSURE: one FREE connect hour, per account, in
                 September 2003
NEWS 13
        AUG 15
                 TEMA: one FREE connect hour, per account, in
                 September 2003
NEWS 14
        AUG 18
                 Data available for download as a PDF in RDISCLOSURE
NEWS 15
        AUG 18
                 Simultaneous left and right truncation added to PASCAL
        AUG 18
NEWS 16
                 FROSTI and KOSMET enhanced with Simultaneous Left and Righ
                 Truncation
        AUG 18
NEWS 17
                 Simultaneous left and right truncation added to ANABSTR
NEWS 18
        SEP 22
                DIPPR file reloaded
NEWS EXPRESS
             April 4 CURRENT WINDOWS VERSION IS V6.01a, CURRENT
              MACINTOSH VERSION IS V6.0b(ENG) AND V6.0Jb(JP),
              AND CURRENT DISCOVER FILE IS DATED 01 APRIL 2003
NEWS HOURS
              STN Operating Hours Plus Help Desk Availability
NEWS INTER
              General Internet Information
NEWS LOGIN
              Welcome Banner and News Items
              Direct Dial and Telecommunication Network Access to STN
NEWS PHONE
NEWS WWW
              CAS World Wide Web Site (general information)
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10085368.2 Page 2

FILE 'HOME' ENTERED AT 13:06:34 ON 24 SEP 2003

=> file registry
COST IN U.S. DOLLARS

SINCE FILE TOTAL ENTRY SESSION 0.42 0.42

FULL ESTIMATED COST

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STRUCTURE FILE UPDATES: 23 SEP 2003 HIGHEST RN 591719-82-3 DICTIONARY FILE UPDATES: 23 SEP 2003 HIGHEST RN 591719-82-3

TSCA INFORMATION NOW CURRENT THROUGH JULY 14, 2003

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Crossover limits have been increased. See HELP CROSSOVER for details.

Experimental and calculated property data are now available. See HELP PROPERTIES for more information. See STNote 27, Searching Properties in the CAS Registry File, for complete details: http://www.cas.org/ONLINE/STN/STNOTES/stnotes27.pdf

=> Uploading 10085368.2

L1 STRUCTURE UPLOADED

=> d ll L1 HAS NO ANSWERS L1 STR

Patel

9/24/2003>

Structure attributes must be viewed using STN Express query preparation.

=> s 11

SAMPLE SEARCH INITIATED 13:07:46 FILE 'REGISTRY' SAMPLE SCREEN SEARCH COMPLETED - 1007 TO ITERATE

99.3% PROCESSED 1000 ITERATIONS INCOMPLETE SEARCH (SYSTEM LIMIT EXCEEDED)

SEARCH TIME: 00.00.01

FULL FILE PROJECTIONS: ONLINE **COMPLETE**

BATCH **COMPLETE**

PROJECTED ITERATIONS:

18237 TO 22043 22 TO 420 11 ANSWERS

PROJECTED ANSWERS: 22 TO

L2 11 SEA SSS SAM L1

=> s l1 sss full

FULL SEARCH INITIATED 13:07:53 FILE 'REGISTRY'
FULL SCREEN SEARCH COMPLETED - 19899 TO ITERATE

100.0% PROCESSED 19899 ITERATIONS 210 ANSWERS

SEARCH TIME: 00.00.01

L3 210 SEA SSS FUL L1

=> file caplus

COST IN U.S. DOLLARS SINCE FILE TOTAL ENTRY SESSION

FULL ESTIMATED COST 148.15 148.57

FILE 'CAPLUS' ENTERED AT 13:08:00 ON 24 SEP 2003 USE IS SUBJECT TO THE TERMS OF YOUR STN CUSTOMER AGREEMENT.

Page 4

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FILE COVERS 1907 - 24 Sep 2003 VOL 139 ISS 13 FILE LAST UPDATED: 23 Sep 2003 (20030923/ED)

This file contains CAS Registry Numbers for easy and accurate substance identification.

=> s 13

L4 309 L3

=> s 14 and phenyl

L5 53 L4 AND PHENYL

=> s 14 and pyridine

L6 37 L4 AND PYRIDINE

=> s 14 and pyridazine

L7 0 L4 AND PYRIDAZINE

=> s 14 and pyrimidine

L8 0 L4 AND PYRIMIDINE

=> s 14 anf furan

MISSING OPERATOR L4 ANF

The search profile that was entered contains terms or nested terms that are not separated by a logical operator.

=> s 14 and furan

L9 1 L4 AND FURAN

=> s 14 and thien

L10 1 L4 AND THIEN

=> s 15 and 16

L11 6 L5 AND L6

=> s 19 and 110

L12 0 L9 AND L10

=> d his

(FILE 'HOME' ENTERED AT 13:06:34 ON 24 SEP 2003)

FILE 'REGISTRY' ENTERED AT 13:07:23 ON 24 SEP 2003

Patel

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10085368.2 Page 5
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L2
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L3
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     FILE 'CAPLUS' ENTERED AT 13:08:00 ON 24 SEP 2003
L4
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L5
L6
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L7
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L8
              0 S L4 AND PYRIMIDINE
L9
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L10
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L111
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=> d l4 fbib hitstr abs total
     ANSWER 1 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN
L4
     2003:570785 CAPLUS
AN
DN
     139:122461
ΤI
     Cosmetic or dermatological preparations containing antioxidants and boron
     compounds for preventing damages to skin caused by peroxides
     Jentzsch, Axel; Haremza, Sylke; Wagenblast, Gerhard
IN
     BASF Aktiengesellschaft, Germany
PΑ
     PCT Int. Appl., 45 pp.
SO
     CODEN: PIXXD2
DT
     Patent
LΑ
    German
FAN.CNT 1
                 KIND DATE
    PATENT NO.
                                         APPLICATION NO. DATE
    WO 2003059312 A2 20030724 WO 2003-EP14 20030103
PΤ
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             CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, ES, FI, GB, GD, GE, GH,
             GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR,
             LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NO, NZ, OM, PH,
             PL, PT, RO, RU, SC, SD, SE, SG, SK, SL, TJ, TM, TN, TR, TT, TZ,
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             NL, PT, SE, SI, SK, TR, BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW,
            ML, MR, NE, SN, TD, TG
                                           DE 2002-10202065A 20020118
                                           DE 2002-10203414A 20020128
     DE 10202065
                            20030724
                                           DE 2002-10202065 20020118
OS
     MARPAT 139:122461
IT
     20631-84-9
     RL: COS (Cosmetic use); BIOL (Biological study); USES (Uses)
        (cosmetic or dermatol. prepns. contg. antioxidants and boron compds.
        for preventing damages to skin caused by peroxides)
RN
     20631-84-9 CAPLUS
CN
     Borinic acid, bis(2,4,6-trimethylphenyl) - (9CI) (CA INDEX NAME)
```

9/24/2003>

The invention relates to cosmetic or dermatol. prepns. that are characterized by having a content of: (a) at least one antioxidant that acts as an O- or C-radical scavenger, and; (b) at least one org., boron-contg. compd. that reduces the peroxides or hydroperoxides to the corresponding alcs. without forming active radical in subsequent stages. Thus a soft skin fluid contained (wt./wt.%): Ceteareth-6 and stearyl alc. 2.50; Ceteareth-25 2.50; hydrogenated coco glycerides 1.50; PEG-40 dodecyl glycol copolymer 3.00; dimethicone 3.00; penethyl dimethicone 2.00; cyclodimethicone 1.00; cetearyl octanoate 5.00; avocado oil 1.00; sweet almond oil 2.00; wheat germ oil 0.80; panthenol 1.00; phytantriol 0.20; tocopheryl acetate 0.30; propylene glycol 5.00; benzeneboronic acid 1.00; sodium ascorbyl phosphate 2.00; perfume, preservative q.s., water to 100.

- L4 ANSWER 2 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN
- AN 2003:463809 CAPLUS
- DN 139:180104
- TI Synthesis and Reactivity of (C6F5)3B-N-Heterocycle Complexes. 1. Generation of Highly Acidic sp3 Carbons in Pyrroles and Indoles
- AU Guidotti, Simona; Camurati, Isabella; Focante, Francesca; Angellini, Luca; Moscardi, Gilberto; Resconi, Luigi; Leardini, Rino; Nanni, Daniele; Mercandelli, Pierluigi; Sironi, Angelo; Beringhelli, Tiziana; Maggioni, Daniela
- CS Centro Ricerche G. Natta, Basell Polyolefins, Ferrara, I-44100, Italy
- SO Journal of Organic Chemistry (2003), 68(14), 5445-5465 CODEN: JOCEAH; ISSN: 0022-3263
- PB American Chemical Society
- DT Journal
- LA English
- IT 155962-45-1P, (Aqua)tris(pentafluorophenyl)boron
 RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT
 (Reactant or reagent)

(nitrogen heterocycle coordination; prepn. and acidity of tris(pentafluorophenyl)borane adducts of nitrogen heterocycles)

- RN 155962-45-1 CAPLUS
- CN Boron, aquatris(pentafluorophenyl)-, (T-4)- (9CI) (CA INDEX NAME)

IT 578731-99-4 578732-00-0

RL: FMU (Formation, unclassified); PRP (Properties); FORM (Formation, nonpreparative)

(optimized geometry, total energy; prepn. and acidity of tris(pentafluorophenyl)borane adducts of nitrogen heterocycles)

RN 578731-99-4 CAPLUS

CN Borate(1-), hydroxytris(pentafluorophenyl)-, (T-4)-, hydrogen, compd. with 3H-indole (1:1) (9CI) (CA INDEX NAME)

CM 1

CRN 147892-17-9

CMF C18 H B F15 O . H

CCI CCS

$$F \longrightarrow C \longrightarrow B \longrightarrow F \longrightarrow F$$

$$F \longrightarrow F \longrightarrow F$$

$$F \longrightarrow F$$

$$F \longrightarrow F$$

$$F \longrightarrow F$$

● H+

CM 2

CRN 271-26-1 CMF C8 H7 N

RN 578732-00-0 CAPLUS

CN Borate(1-), hydroxytris(pentafluorophenyl)-, (T-4)-, hydrogen, compd. with 2H-pyrrole (1:1) (9CI) (CA INDEX NAME)

CM 1

CRN 147892-17-9

CMF C18 H B F15 O . H

CCI CCS

$$F \longrightarrow F \longrightarrow F \longrightarrow F$$

$$F \longrightarrow F \longrightarrow F$$

$$F \longrightarrow F$$

● H+

CM 2

CRN 287-97-8 CMF C4 H5 N



AΒ The reaction of pyrroles and indoles with B(C6F5)3 and BCl3 produces 1:1 complexes contg. highly acidic sp3 carbons and B--N+ betaine fragment, which are the results of NH-proton migration to adjacent carbon atom. Reaction of B(C6F5)3 with 2-R1-3-R3-5-R2-1H-pyrrole gave B--N+ betaine 2-R1-3-R3-5-R2-2H-pyrrole-1-[tris(pentafluorophenyl)borane] adducts (1, 3-5; R1, R2, R3: H, H, H; Me, Me, H; H, Me Me; H, Et, H), the same reaction of 2-R4-3-R5-5-R6-1H-indole afforded 2-R4-3-R5-5-R6-3H-indole-1-[tris(pentafluorophenyl)borane] (2, 7-9, 11; R4, R5, R6: H, H, H; Me, H, H; H, Me, H; H, H, OMe; H, H, Cl). 3-[Tris(pentafluorophenyl)borane]-1Himidazole (17) and 2-[tris(pentafluorophenyl)borane]-1H-pyrazole (18) adducts were also prepd., the latter gave deprotonation product, the triethylammonium [tris(pentafluorophenyl)](1H-pyrazol-1-yl)borate (18a). The mechanism of the new formal N-to-C hydrogen shift is discussed. For the complexes 2, 4, 5, 7 and 8, restricted rotation around the B-N bond and/or the B-C bonds was obsd. by NMR techniques, and rotational barriers for 2, 4 and 7 were calcd. from exptl. data. The acidity of the sp3 carbons in these complexes is shown by their ability to protonate NEt3, with formation of pyrrolyl- and indolyl-borate ammonium salts. The driving force for this reaction is given by the restoration of the aromaticity of the heterocycle.

RE.CNT 56 THERE ARE 56 CITED REFERENCES AVAILABLE FOR THIS RECORD ALL CITATIONS AVAILABLE IN THE RE FORMAT

L4 ANSWER 3 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

AN 2003:404013 CAPLUS

DN 139:133072

TI Ring-Opening Reactions of Nonactivated Aziridines Catalyzed by Tris(pentafluorophenyl)borane

Patel

10085368.2

Page 9

AU Watson, Iain D. G.; Yudin, Andrei K.

CS Davenport Research Laboratories, Department of Chemistry, The University of Toronto, Toronto, ON, M5S 3H6, Can.

SO Journal of Organic Chemistry (2003), 68(13), 5160-5167 CODEN: JOCEAH; ISSN: 0022-3263

PB American Chemical Society

DT Journal

LA English

IT 568599-28-0P

RL: PRP (Properties); SPN (Synthetic preparation); PREP (Preparation) (crystal structure of aminocyclohexanol-tris(pentafluorophenyl)borane complex, prepd. as intermediate in ring-opening of nonactivated aziridines)

RN 568599-28-0 CAPLUS

CN Boron, aquatris(pentafluorophenyl)-, (T-4)-, compd. with rel-(1R,2R)-2-[(phenylmethyl)amino]cyclohexanol (1:1) (9CI) (CA INDEX NAME)

CM 1

CRN 155962-45-1 CMF C18 H2 B F15 O CCI CCS

$$F \xrightarrow{F} GH2 \xrightarrow{F} F$$

$$F \xrightarrow{F} F \xrightarrow{F} F$$

CM 2

CRN 40571-86-6 CMF C13 H19 N O

Relative stereochemistry.

IT 312640-05-4 568599-29-1

RL: FMU (Formation, unclassified); FORM (Formation, nonpreparative)

Patel

9/24/2003>

(mechanistic studies of tris(pentafluorophenyl)borane catalyzed ring-opening of nonactivated aziridines with amines or benzenethiol)

RN 312640-05-4 CAPLUS

CN Boron, aquatris(pentafluorophenyl)-, monohydrate, (T-4)- (9CI) (CA INDEX NAME)

● H₂O

RN 568599-29-1 CAPLUS

CN Boron, aquatris(pentafluorophenyl)-, (T-4)-, compd. with 7-(phenylmethyl)-7-azabicyclo[4.1.0]heptane (1:1), monohydrate (9CI) (CAINDEX NAME)

CM 1

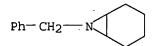
CRN 155962-45-1 CMF C18 H2 B F15 O CCI CCS

$$F \xrightarrow{F} C^{-} \xrightarrow{OH_2} F \xrightarrow{F} F$$

$$F \xrightarrow{F} F \xrightarrow{F} F$$

CM 2

CRN 24417-01-4 CMF C13 H17 N



AB The ring-opening reactions of nonactivated aziridines with amine nucleophiles are efficiently catalyzed by tris(pentafluorophenyl)borane leading to the corresponding trans-1,2-diamines in high yields. A mechanistic investigation of the reaction suggests that in situ formed [(C6F5)3B(OH2)].cntdot.H2O catalyzes the opening through a Bronsted acid manifold.

RE.CNT 79 THERE ARE 79 CITED REFERENCES AVAILABLE FOR THIS RECORD ALL CITATIONS AVAILABLE IN THE RE FORMAT

L4 ANSWER 4 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

AN 2003:324011 CAPLUS

DN 139:62062

TI Protonation and Reactivity towards Carbon Dioxide of the Mononuclear Tetrahedral Zinc and Cobalt Hydroxide Complexes, [TpBut,Me]ZnOH and [TpBut,Me]CoOH: Comparison of the Reactivity of the Metal Hydroxide Function in Synthetic Analogues of Carbonic Anhydrase

AU Bergquist, Catherine; Fillebeen, Tauqir; Morlok, Melissa M.; Parkin, Gerard

CS Department of Chemistry, Columbia University, New York, NY, 10027, USA

SO Journal of the American Chemical Society (2003), 125(20), 6189-6199 CODEN: JACSAT; ISSN: 0002-7863

PB American Chemical Society

DT Journal

LA English

IT 547766-78-9P 547766-79-0P

RL: PRP (Properties); SPN (Synthetic preparation); PREP (Preparation) (prepn. and crystal structure)

RN 547766-78-9 CAPLUS

CN Zinc(1+), aqua[tris[3-(1,1-dimethylethyl)-5-methyl-1H-pyrazolato-.kappa.N1]hydroborato(1-)-.kappa.N2',.kappa.N2',.kappa.N2'']-, (T-4)-, (T-4)-hydroxytris(pentafluorophenyl)borate(1-), compd. with benzene (2:1) (9CI) (CA INDEX NAME)

CM 1

CRN 71-43-2 CMF C6 H6



CM 2

CRN 243121-77-9

CMF C24 H42 B N6 O Zn . C18 H B F15 O

CM 3

CRN 243121-76-8

10085368.2

Page 12

CMF C24 H42 B N6 O Zn CCI CCS

Me
$$N = N$$
 $N = N$ N

CM 4

CRN 148657-98-1 CMF C18 H B F15 O CCI CCS

RN 547766-79-0 CAPLUS

CN Cobalt(1+), aqua[tris[3-(1,1-dimethylethyl)-5-methyl-1H-pyrazolato-.kappa.N1]hydroborato(1-)-.kappa.N2',.kappa.N2',.kappa.N2'']-, (T-4)-, (T-4)-hydroxytris(pentafluorophenyl)borate(1-), compd. with benzene (2:1) (9CI) (CA INDEX NAME)

CM 1

CRN 71-43-2 CMF C6 H6



CM 2

CRN 547766-75-6 CMF C24 H42 B Co N6 O . C18 H B F15 O

CM 3

CRN 547766-74-5 CMF C24 H42 B CO N6 O CCI CCS

Me N N N
$$Co^{2+}$$
 OH2

Me N Bu-t

Me Bu-t

CM 4

CRN 148657-98-1 CMF C18 H B F15 O CCI CCS

IT 243121-77-9P 547766-75-6P

RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent) (prepn. and reaction with CO2, Bu4NI and NEt3 as model for carbonic anhydrase)

RN 243121-77-9 CAPLUS

CN Zinc(1+), aqua[tris[3-(1,1-dimethylethyl)-5-methyl-1H-pyrazolato-.kappa.N1]hydroborato(1-)-.kappa.N2,.kappa.N2',.kappa.N2'']-, (T-4)-, (T-4)-hydroxytris(pentafluorophenyl)borate(1-) (9CI) (CA INDEX NAME)

CM 1

CRN 243121-76-8 CMF C24 H42 B N6 O Zn CCI CCS

Me N N N
$$2n^{2+}$$
 OH₂

Me N Bu-t

Me Bu-t

Bu-t

CM 2

CRN 148657-98-1 CMF C18 H B F15 O CCI CCS

RN 547766-75-6 CAPLUS

CN Cobalt(1+), aqua[tris[3-(1,1-dimethylethyl)-5-methyl-1H-pyrazolato-.kappa.N1]hydroborato(1-)-.kappa.N2,.kappa.N2',.kappa.N2'']-, (T-4)-, (T-4)-hydroxytris(pentafluorophenyl)borate(1-) (9CI) (CA INDEX NAME)

CM 1

CRN 547766-74-5

CMF C24 H42 B Co N6 O

CCI CCS

Me N N N
$$Co^{2+}$$
 OH2

Me N Bu-t

Me Bu-t

Bu-t

CM 2

CRN 148657-98-1 CMF C18 H B F15 O CCI CCS

$$F \xrightarrow{F} C^{-} \xrightarrow{OH_2} F \xrightarrow{F} F$$

$$F \xrightarrow{F} F \xrightarrow{F} F$$

AB The tris(3-tert-butyl-5-methylpyrazolyl)hydroborato Zn hydroxide complex

Patel

[TpBut,Me] ZnOH is protonated by (C6F5)3B(OH2) to yield the aqua deriv. {[TpBut,Me]Zn(OH2)}[HOB(C6F5)3], which was structurally characterized by x-ray diffraction, thereby demonstrating that protonation results in a lengthening of the Zn-O bond by .apprx. 0.1 .ANG.. The protonation is reversible, and treatment of {[TpBut,Me]Zn(OH2)}+ with Et3N regenerates [TpBut, Me] ZnOH. Consistent with the notion that the catalytic hydration of CO2 by carbonic anhydrase requires deprotonation of the coordinated H2O mol., { [TpBut, Me] Zn(OH2) } + is inert towards CO2, whereas [TpBut, Me] ZnOH is in rapid equil. with the bicarbonate complex [TpBut,Me] ZnOC(0)OH under comparable conditions. The Co hydroxide complex [TpBut,Me]CoOH is likewise protonated by (C6F5)3B(OH2) to yield the aqua deriv. $\{[TpBut,Me]Co(OH2)\}[HOB(C6F5)3],$ which is isostructural with the Zn complex. The aqua complexes $\{[TpBut,Me]M(OH2)\}[HOB(C6F5)3]$ (M = Zn, Co) exhibit a H bonding interaction between the metal aqua and B hydroxide moieties. This H bonding interaction may be viewed as analogous to that between the aqua ligand and Thr-199 at the active site of carbonic anhydrase. In addn. to the structural similarities between the Zn and Co complexes, [TpBut,Me] ZnOH and [TpBut,Me] CoOH, and between $\{[TpBut,Me]Zn(OH2)\}+ and \{[TpBut,Me]Co(OH2)\}+, DFT (B3LYP) calcns.$ demonstrate that the pKa value of {[Tp]Zn(OH2)}+ is similar to that of {[Tp]Co(OH2)}+. These similarities are in accord with the observation that CoII is a successful substitute for ZnII in carbonic anhydrase. The Co hydroxide [TpBut,Me]CoOH reacts with CO2 to give the bridging carbonate complex { [TpBut, Me] Co}2(.mu.-.eta.1,.eta.2-CO3). The coordination mode of the carbonate ligand in this complex, which is bidentate to one Co center and unidentate to the other, is in contrast to that in the Zn counterpart {[TpBut,Me]Zn}2(.mu.-.eta.1,.eta.1-CO3), which bridges in a unidentate manner to both Zn centers. This difference in coordination modes concurs with the suggestion that a possible reason for the lower activity of CoII-carbonic anhydrase is assocd. with enhanced bidentate coordination of bicarbonate inhibiting its displacement.

RE.CNT 97 THERE ARE 97 CITED REFERENCES AVAILABLE FOR THIS RECORD ALL CITATIONS AVAILABLE IN THE RE FORMAT

```
L4
     ANSWER 5 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN
     2003:319726 CAPLUS
ΑN
DN
     138:338291
ΤI
     Preparation of arylboronic acid derivatives and esters thereof as
     intracellular calcium concentration increase inhibitors
ΙN
     Mikoshiba, Katsuhiko; Iwasaki, Hirohide; Maruyama, Takayuki; Hamano,
     Shinichi
PA
    Ono Pharmaceutical Co., Ltd., Japan
SO
     PCT Int. Appl., 128 pp.
     CODEN: PIXXD2
DT
     Patent
LΑ
     Japanese
FAN.CNT 1
     PATENT NO.
                     KIND
                           DATE
                                          APPLICATION NO.
                                                            DATE
                                           -----
PΙ
     WO 2003033002
                     A1
                            20030424
                                          WO 2002-JP10534 20021010
        W: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN,
             CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, ES, FI, GB, GD, GE, GH,
             GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KR, KZ, LC, LK, LR, LS,
             LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NO, NZ, OM, PH, PL,
             PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TN, TR, TT, TZ, UA,
             UG, US, UZ, VC, VN, YU, ZA, ZM, ZW, AM, AZ, BY, KG, KZ, MD, RU,
         RW: GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZM, ZW, AT, BE, BG,
```

CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, SK, TR, BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG

JP 2001-313402 A 20011011

OS MARPAT 138:338291

IT 515157-07-0P 515157-09-2P 515157-11-6P

515157-13-8P 515157-15-0P 515157-17-2P

515157-19-4P 515157-21-8P 515157-23-0P

515157-25-2P 515157-27-4P 515157-29-6P

515157-31-0P 515157-33-2P 515157-35-4P

515157-37-6P 515157-38-7P 515157-40-1P

515157-42-3P 515157-44-5P 515157-47-8P

515157-50-3P

RL: PAC (Pharmacological activity); SPN (Synthetic preparation); THU (Therapeutic use); BIOL (Biological study); PREP (Preparation); USES (Uses)

(prepn. of arylboronic acid derivs. and esters thereof as intracellular calcium concn. increase inhibitors as preventives and/or remedies for diseases)

RN 515157-07-0 CAPLUS

CN Borinic acid, (3-chloro-4-methylphenyl)[4-[(phenylmethoxy)methyl]phenyl](9CI) (CA INDEX NAME)

RN 515157-09-2 CAPLUS

CN Borinic acid, [2-[(diethylamino)carbonyl]phenyl]phenyl- (9CI) (CA INDEX NAME)

RN 515157-11-6 CAPLUS

CN Borinic acid, (4-chlorophenyl)[4-[(phenylmethoxy)methyl]phenyl]- (9CI) (CA INDEX NAME)

RN 515157-13-8 CAPLUS

CN Borinic acid, (4-chlorophenyl)[4-[[(4-methoxyphenyl)methoxy]methyl]phenyl](9CI) (CA INDEX NAME)

RN 515157-15-0 CAPLUS

CN Borinic acid, (4-chlorophenyl) [4-[[(1,2,3,4-tetrahydro-1-naphthalenyl)oxy]methyl]phenyl]- (9CI) (CA INDEX NAME)

RN 515157-17-2 CAPLUS

CN Borinic acid, (4-chlorophenyl)[4-[(2-phenylethoxy)methyl]phenyl]- (9CI) (CA INDEX NAME)

$$\begin{array}{c|c} \text{C1} & \text{OH} & \text{CH}_2\text{--}\text{O--}\text{CH}_2\text{--}\text{CH}_2\text{--}\text{Ph} \\ \\ & \text{B} & \end{array}$$

RN 515157-19-4 CAPLUS

CN Borinic acid, (4-chlorophenyl)[4-[(cyclohexyloxy)methyl]phenyl]- (9CI) (CA INDEX NAME)

RN 515157-21-8 CAPLUS

CN Borinic acid, [4-(butoxymethyl)phenyl](4-chlorophenyl)- (9CI) (CA INDEX NAME)

RN 515157-23-0 CAPLUS

CN Borinic acid, [1,1'-biphenyl]-4-yl[4-[(phenylmethoxy)methyl]phenyl]- (9CI) (CA INDEX NAME)

$$\begin{array}{c|c} \text{OH} & \text{OH} \\ \hline \\ \text{Ph-CH}_2\text{-O-CH}_2 & \text{Ph} \end{array}$$

RN 515157-25-2 CAPLUS

CN Borinic acid, (4-chlorophenyl)[3-[(phenylmethoxy)methyl]phenyl]- (9CI) (CA.INDEX NAME)

RN 515157-27-4 CAPLUS

CN Borinic acid, (3,5-dichlorophenyl)[4-[(phenylmethoxy)methyl]phenyl]- (9CI) (CA INDEX NAME)

RN 515157-29-6 CAPLUS

CN Borinic acid, (4-bromophenyl) [4-[(phenylmethoxy)methyl]phenyl]- (9CI) (CA

INDEX NAME)

RN 515157-31-0 CAPLUS

CN Borinic acid, (4-chlorophenyl)[4-[2-[[(phenylamino)carbonyl]oxy]ethyl]phen yl]- (9CI) (CA INDEX NAME)

RN 515157-33-2 CAPLUS

CN Carbonic acid, 2-[4-[(4-chlorophenyl)hydroxyboryl]phenyl]ethyl methyl ester (9CI) (CA INDEX NAME)

RN 515157-35-4 CAPLUS

CN Carbamic acid, (2-methyl-4-nitrophenyl)-, 2-[4-[(4-chlorophenyl)hydroxyboryl]phenyl]ethyl ester (9CI) (CA INDEX NAME)

RN 515157-37-6 CAPLUS

CN 1,3-Benzenedicarboxylic acid, 5-[[[2-[4-[(4-chlorophenyl)hydroxyboryl]phen yl]ethoxy]carbonyl]amino]-, dimethyl ester (9CI) (CA INDEX NAME)

$$\begin{array}{c} \text{MeO-C} \\ \text{MeO-C} \\ \text{O} \\ \text{NH-C-O-CH}_2\text{-CH}_2 \\ \end{array}$$

RN 515157-38-7 CAPLUS

CN L-Valine, N-[[2-[4-[(4-chlorophenyl)hydroxyboryl]phenyl]ethoxy]carbonyl]-, ethyl ester (9CI) (CA INDEX NAME)

Absolute stereochemistry.

RN 515157-40-1 CAPLUS

CN Borinic acid, [oxybis(methylene-2,1-phenylene)]bis[phenyl- (9CI) (CA INDEX NAME)

RN 515157-42-3 CAPLUS

CN Borinic acid, [1,4-butanediylbis(oxy-4,1-phenylene)]bis[phenyl- (9CI) (CA INDEX NAME)

RN · 515157-44-5 CAPLUS

CN Borinic acid, [oxybis(methylene-4,1-phenylene)]bis[phenyl- (9CI) (CF INDEX NAME)

$$HO-B$$
 Ph
 Ph
 Ph
 Ph
 Ph

RN 515157-47-8 CAPLUS

CN Borinic acid, bis(3-chloro-4-methylphenyl) - (9CI) (CA INDEX NAME)

RN 515157-50-3 CAPLUS

CN 2-Propenoic acid, 3,3'-[(hydroxyborylene)di-4,1-phenylene]bis-, dimethyl ester, (2E,2'E)- (9CI) (CA INDEX NAME)

Double bond geometry as shown.

GI

AB Disclosed are intracellular calcium concn. increase inhibitors contg. as the active ingredient boron compds. represented by the following general formula (I) [R1 = (un)substituted C1-3 aminoalkyl, C1-6 alkyl or C2-6 alkenyl substituted by 5- or 6-membered monocyclic heterocyclyl or C5 or 6 monocyclic carbocyclyl, CHR5R6, 5,6,7,8-tetrahydroquinolin-8-yl (wherein R5, R6 = (un)substituted C4-5 monocyclic carbocyclyl, C5 or 6 monocyclic carbocyclyl, or C1-6 alkyl or C2-6 alkenyl substituted by one of these groups); G = HO, (un)substituted C5-10 monocyclic or bicyclic carbocyclyl or 5 to 10-membered monocyclic or bicyclic heterocyclyl; Cyc2 =

Patel

(un) substituted C5-10 monocyclic or bicyclic carbocyclyl or 5 to 10-membered monocyclic or bicyclic heterocyclyl; E = a single bond, C1-4 alkylene optionally substituted C5 or 6 monocyclic carbocyclyl; provided that (2-aminoethoxy)diphenylborane] or nontoxic salts thereof. compds. inhibit the increase in intracellular calcium calcium by inhibiting the release of endogenous calcium or the influx of capacitive calcium and are useful as preventives and/or remedies for platelet aggregation, ischemic diseases in the heart or brain, immune deficiency, allergic diseases, bronchial asthma, hypertension, cerebrovascular twitch, various renal diseases, pancreatitis, and Alzheimer's disease. Thus, a soln. of 100 mg bis(3-chloro-4-methylphenyl)boronic acid and 52 mg N-cyclohexylethanolamine in 2 mL ethanol was stirred at room temp. overnight to give bis(3-chloro-4-methylphenyl)boronic acid 2-cyclohexylaminoethyl ester (II). Bis[4-[(2aminoethoxy)phenylboryl]benzyl] ether showed IC50 of 0.059 .mu.M for inhibiting the increase in intracellular calcium concn. in chicken-derived DT40 cultured cells lacking in IP3 receptor and also depleted in calcium by treatment with thapsigargin (calcium ion pump inhibitor for endoplasmic reticulum) when the cells were exposed to calcium chloride soln. A tablet and an ampule formulation contq. II were described.

RE.CNT 13 THERE ARE 13 CITED REFERENCES AVAILABLE FOR THIS RECORD ALL CITATIONS AVAILABLE IN THE RE FORMAT

L4 ANSWER 6 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

AN 2003:309245 CAPLUS

DN 138:321018

TI Preparation of tert-amyloxyhalogenobenzenes, tert-amyloxycyanobiphenyl, and cyanohydroxybiphenyl

IN Nakamura, Akira; Masaoka, Shin

PA Hokko Chemical Industry Co., Ltd., Japan

SO Jpn. Kokai Tokkyo Koho, 12 pp.

CODEN: JKXXAF

DT Patent

LA Japanese

FAN.CNT 1

PΙ

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 2003119175	A2	20030423	JP 2001-316684	20011015
			JP 2001-316684	20011015

OS MARPAT 138:321018

IT 512833-77-1

RL: RCT (Reactant); RACT (Reactant or reagent)

(in prepn. of amyloxycyanobiphenyl; prepn. of cyanohydroxybiphenyl by reaction of amyloxyhalogenobenzenes with Mg and borates, condensed with halogenobenzonitrile, and hydrolysis of amyloxycyanobiphenyl)

RN 512833-77-1 CAPLUS

CN Borinic acid, bis[4-(1,1-dimethylpropoxy)phenyl]- (9CI) (CA INDEX NAME)

AB HOC6H4C6H4CN is prepd. by reaction of XC6H4CN (X = Cl, Br, I) with

Patel

(MeCH2CMe2OC6H4)3-nB(OH)n (I; n = 0-2) in the presence of transition metal catalysts and bases and hydrolysis of MeCH2CMe2OC6H4C6H4CN under acidic condition. I (n = 0-2) is prepd. by reaction of HOC6H4X (X = Cl, Br, I) with 2-methyl-2-butene, treatment with Mg, and reaction with (RO)3B (R = Cl-6 alkyl), and hydrolysis. 4-Tert-amyloxychlorobenzene was treated with Mg in the presence of EtBr in THF-PhMe at 70.degree. for 5 h and reacted with tri-n-hexyl borate at 35-45.degree. for 2 h to give $4,4^{+}$ -di(tert-amyloxyphenyl)borinic acid, which was treated with 4-ClC6H4CN in the presence of K3PO4 and 1,4-bis(diphenylphosphino)butanenickel(II) chloride under reflux for 2 h to give $98.7 \% 4^{+}$ -tert-amyloxy-4-cyanobiphenyl, which was hydrolyzed with H2SO4 in THF at 30.degree. for 4 h to give 94.4 % 4-cyano-4'-hydroxybiphenyl.

- L4 ANSWER 7 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN
- AN 2003:193808 CAPLUS
- DN 138:354016
- TI Bis(pentafluorophenyl)borinic Acid: a Cyclic Trimer in the Solid State and a Monomer, with Hindered Rotation around the B-OH Bond, in Solution
- AU Beringhelli, Tiziana; D'Alfonso, Giuseppe; Donghi, Daniela; Maggioni, Daniela; Mercandelli, Pierluigi; Sironi, Angelo
- CS Dipartimento di Chimica Inorganica Metallorganica e Analitica, Milan, 20133, Italy
- SO Organometallics (2003), 22(8), 1588-1590 CODEN: ORGND7; ISSN: 0276-7333
- PB American Chemical Society
- DT Journal
- LA English
- IT 2118-02-7, Bis(pentafluorophenyl)borinic acid
 RL: CPS (Chemical process); PEP (Physical, engineering or chemical
 process); PRP (Properties); RCT (Reactant); PROC (Process); RACT (Reactant
 or reagent)
 - (bis (pentafluorophenyl) borinic acid as cyclic trimer in solid state and a monomer, with hindered rotation around the boron-hydroxy bond, in soln.)
- RN 2118-02-7 CAPLUS
- CN Borinic acid, bis(pentafluorophenyl) (7CI, 8CI, 9CI) (CA INDEX NAME)

- AB The title mol. in the solid state exists as a cyclic trimer, with B-O(H)-B bridges and a cyclohexane-like structure (C2 twist-boat conformation); dissoln. in toluene-d8 affords the B(C6F5)2OH monomer, in which the low-temp. 19F NMR data reveal restricted rotation of the OH substituent around the Ar2B-OH bond (Ea = 39 kJ mol-1), as a result of the partial double-bond character of this interaction. The crystal structure of trimer was detd.
- RE.CNT 41 THERE ARE 41 CITED REFERENCES AVAILABLE FOR THIS RECORD ALL CITATIONS AVAILABLE IN THE RE FORMAT

```
L4
    ANSWER 8 OF 309 CAPLUS
                              COPYRIGHT 2003 ACS on STN
AN
     2003:150618 CAPLUS
DN
     138:190256
ΤI
     Process for the preparation or aryl and alkyl boron compounds in micro
     Koch, Manfred; Wehle, Detlef; Scherer, Stefan; Forstinger, Klaus; Meudt,
IN
    Andreas; Hessel, Volker; Werner, Bernd; Loewe, Holger
     Clariant Gmbh, Germany
PA
SO
     Eur. Pat. Appl., 12 pp.
     CODEN: EPXXDW
DT
     Patent
LΑ
    German
FAN.CNT 1
     PATENT NO.
                     KIND DATE
                                          APPLICATION NO. DATE
                     ____
                           -----
                                          -----
PΙ
     EP 1285924
                           20030226
                                          EP 2002-16149 20020720
                     A1
        R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT,
            IE, SI, LT, LV, FI, RO, MK, CY, AL, TR, BG, CZ, EE, SK
                                          DE 2001-10140857A 20010821
    DE 10140857
                      A1
                            20030306
                                          DE 2001-10140857 20010821
    US 2003100792
                      Α1
                            20030529
                                          US 2002-210807
                                                          20020801
                                          DE 2001-10140857A 20010821
                           20030508
    JP 2003128677
                                          JP 2002-240103
                      A2
                                                            20020821
                                          DE 2001-10140857A 20010821
IT
    2622-89-1P, Diphenylborinic acid
    RL: SPN (Synthetic preparation); PREP (Preparation)
        (synthesis of trialkyl- and triaryl-substituted boranes, boronic acids,
        and tetraalkylborates in flow-through reactors))
RN
     2622-89-1 CAPLUS
    Borinic acid, diphenyl- (6CI, 7CI, 8CI, 9CI) (CA INDEX NAME)
CN
   Ph
Ph-B-OH
    Manuf. of arylboron and alkylboron compds., of general formulas RnBX3-n
    and R4B- MgY+, as well as RnB(OH)3-n (prepd. by hydrolysis of RnBX3-n),
    halides, R-Mg-Y, and BX3, in which X = F, Cl, Br, I, Cl-5-alkoxy,
```

AB are prepd. from the corresponding arylmagnesium halides and alkylmagnesium N, N-di(C1-5-alkyl) amino, or (C1-5-alkyl) thio; n = 1, 2, or 3; and R = 1C1-6-alkyl, (RO-, RR'N-, Ph-, substituted Ph-, F-, and RS-), and (C1-6-alkyl)-substituted phenyl; and (C1-6-alkyl-, C1-6-alkoxy-, C1-5-thioalkyl-, silyl- F-, Cl-, dialkylamino-, diarylamino-, and alkylarylamino) - substituted Ph, in addn. to heterocycloaryl substituents with one or two heteroatoms (e.g., N, O, or S). The compds. are synthesized in through-flow microreactors in flow channels of diam. 0.25 .mu. to 1.5 mm.

RE.CNT 3 THERE ARE 3 CITED REFERENCES AVAILABLE FOR THIS RECORD ALL CITATIONS AVAILABLE IN THE RE FORMAT

- L4ANSWER 9 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN
- 2003:98804 CAPLUS AN
- DN 138:329264
- ΤI Crystal structure of [1,2-ethylene-1,1'-bis(.eta.5-tetrahydroindenyl)] [hydroxytris(pentafluorophenyl)borato]titanium(III), C38H24BF15OTi
- ΑU Spannenberg, A.; Burlakov, V. V.; Arndt, P.; Baumann, W.; Shut, V. B.;

10085368.2

Page 26

Rosenthal, U.

- CS Institut fur Organische Katalyseforschung an der Universitat Rostock e.V., Rostock, D-18055, Germany
- SO Zeitschrift fuer Kristallographie New Crystal Structures (2002), 217(4), 546-548

CODEN: ZKNSFT; ISSN: 1433-7266

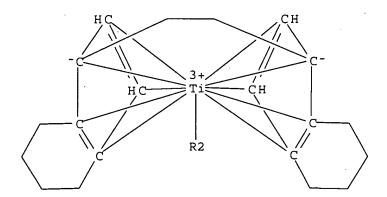
- PB Oldenbourg Wissenschaftsverlag GmbH
- DT Journal
- LA English
- IT 511548-86-0P

RL: PRP (Properties); SPN (Synthetic preparation); PREP (Preparation) (prepn. and crystal structure of)

RN 511548-86-0 CAPLUS

CN Titanium, [rel-(7aR,7'aR)-1,2-ethanediylbis[(1,2,3,3a,7a-.eta.)-4,5,6,7-tetrahydro-1H-inden-1-ylidene]][hydroxytris(pentafluorophenyl)borato(1-)-.kappa.0]-(9CI) (CA INDEX NAME)





PAGE 2-A

$$F \longrightarrow F$$

$$F \longrightarrow F$$

AB The title compd. is monoclinic, space group P21/c, a 8.584(2), b 17.976(4), c 21.964(4) .ANG., .beta. 94.47(3).degree., Z = 4, Rgt(F) = 0.039, wRall(F2) = 0.087, T = 293 K. At. coordinates are given. The mol. structure of the title complex [d(Ti-O) 2.097(3) .ANG., d(O-B) 1.482(4) .ANG.] is comparable with the d in titanocene complex (CpB)CpTi(u-OH)Cl [d(Ti-O) 2.044(1) .ANG., d(O-B) 1.532(3) .ANG.]. Therefore, its structure can be described in the same manner as the donor complex was between the cationic titanium center and the HO-B(C6F5)3 borate anion. The B-O distance of 1.495(5) .ANG. in the title compd. is similar to that found for [Et3NH]+[(C6F5)3BOH]-. The hydrogen atom at the oxygen in the title complex cannot be localized, but because of the described bonding situation there has to was be one. Addnl. the title compd. is paramagnetic.

RE.CNT 12 THERE ARE 12 CITED REFERENCES AVAILABLE FOR THIS RECORD ALL CITATIONS AVAILABLE IN THE RE FORMAT

L4 ANSWER 10 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

AN 2002:955424 CAPLUS

DN 138:40781

TI Triphenyl boron addition product and its use as antifouling agent in coatings

IN Yoshimaru, Masaaki; Kohara, Masanori; Koga, Yuji

PA Yoshitomi Fine Chemicals Ltd., Japan

SO Jpn. Kokai Tokkyo Koho, 20 pp.

CODEN: JKXXAF

DT Patent

LA Japanese

FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
ΡI	JP 2002363187	A2	20021218	JP 2001-211385	20010607
				JP 2001-211385	20010607

9/24/2003>

OS MARPAT 138:40781

Patel

IT 12113-07-4

RL: RCT (Reactant); RACT (Reactant or reagent)
(in prepn. of tri-Ph boron addn. product for antifouling coatings)

RN 12113-07-4 CAPLUS

CN Borate(1-), hydroxytriphenyl-, sodium, (T-4)- (9CI) (CA INDEX NAME)

● Na +

AB The patent relates to antifouling agent useful for fish nets, boat hulls, etc. wherein the antifouling agent is represented by (ph3B)-NH2R1-NH-R2-NH2-(Bph3) and its salts where R1 and R2 are the same or different C1-18 alkyl which may contain oxygen atom. Thus, di(triphenylboran)-3,3'-iminobis(propylamine) adduct prepd. by reacting triphenylboron sodium hydroxide salt soln. and 3,3'-iminobis(propylamine) was used as antifouling agent for fish nets and showed no bio-organism attachment after 6 mo.

L4 ANSWER 11 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

AN 2002:935623 CAPLUS

DN 138:247568

TI Formation and characterization of the first monoalumoxane, LAlO.cntdot.B(C6F5)3

AU Neculai, Dante; Roesky, Herbert W.; Neculai, Ana Mirela; Magull, Jorg; Walfort, Bernhard; Stalke, Dietmar

CS Institut fur Anorganische Chemie Universitat Gottingen, Gottingen, 37077, Germany

SO Angewandte Chemie, International Edition (2002), 41(22), 4294-4296 CODEN: ACIEF5; ISSN: 1433-7851

PB Wiley-VCH Verlag GmbH & Co. KGaA

DT Journal

LA English

OS CASREACT 138:247568

RN 155962-45-1 CAPLUS

CN Boron, aquatris(pentafluorophenyl)-, (T-4)- (9CI) (CA INDEX NAME)

$$F \xrightarrow{F} C \xrightarrow{OH_2} F \xrightarrow{F} F$$

$$F \xrightarrow{F} F$$

$$F \xrightarrow{F} F$$

The reaction of aluminum .beta.-diketiminato complex LAlMe2 [L = Et2NCH2CH2N:C(Me)CHC(Me):NCH2CH2NEt2] with (H2O)B(C6F5)3 in toluene at 0.degree. gave the monoalumoxane, [LAlO.cntdot.B(C6F5)3] (1). The reaction in THF at 55.degree. resulted in the formation of [LAl(C6F5){OB(C6F5)2}] (2). The complexes were characterized by multinuclear NMR spectroscopy, elemental anal. and x-ray structural anal. The crystal structure of 1 shows a tetrahedral Al bonded to the hydroxytriarylborate O atom and 3 N atoms of the diketiminate ligand. The structure of 2 consist of a 5-coordinate Al with the diarylborinate O, aryl C and one diketiminate N atom in the equatorial sites of a trigonal bipyramid. In both solid state structures one arm of the potentially tetradentate .beta.-diketiminate remains uncoordinated, although the soln. 1H NMR spectra show the diketiminate arms to be equiv.

RE.CNT 29 THERE ARE 29 CITED REFERENCES AVAILABLE FOR THIS RECORD ALL CITATIONS AVAILABLE IN THE RE FORMAT

L4 ANSWER 12 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

AN 2002:898289 CAPLUS

DN 138:122686

TI Unsymmetrical 9-Borafluorenes via Low-Temperature C-H Activation of m-Terphenylboranes

AU Wehmschulte, Rudolf J.; Diaz, Armando A.; Khan, Masood A.

CS Department of Chemistry and Biochemistry, University of Oklahoma, Norman, OK, 73019, USA

SO Organometallics (2003), 22(1), 83-92 CODEN: ORGND7; ISSN: 0276-7333

PB American Chemical Society

DT Journal

LA English

Patel

OS CASREACT 138:122686

IT 488838-67-1P

RL: SPN (Synthetic preparation); PREP (Preparation) (prepn., structure, and reactivity of unsym. borafluorenes via low-temp. carbon-hydrogen activation of terphenylboranes)

RN 488838-67-1 CAPLUS

CN Borinic acid, bis[4,4''-bis(1,1-dimethylethyl)[1,1':3',1''-terphenyl]-2'-yl]- (9CI) (CA INDEX NAME)

AB The reaction of 2,6-(4-t-BuC6H4)2C6H3Li and with H2ClB.cntdot.SMe2 or HCl2B.cntdot.SMe2 in hexane soln. afforded the m-terphenyl-substituted unsym. 9-borafluorene 1-(4-tert-butylphenyl)-7-tert-butyl-9-(bis-2,6-(4tert-butylphenyl)phenyl)-9-borafluorene, 1, in good to moderate yields. The related reaction of 2,6-(3,5-Me2C6H3)2C6H3Li with BH2Cl.cntdot.SMe2 or BHCl2.cntdot.SMe2 in toluene soln. gave 1-(3,5-dimethylphenyl)-6,8dimethyl-9-(bis-2,6-(3,5-dimethylphenyl)phenyl)-9-borafluorene, 3. Compds. 1 and 3 are air-stable fluorescent solids. The reactions of 2,6-(2-MeC6H4)2C6H3Li or 2,6-Mes2C6H3Li (which possess either two or no oand o''-hydrogens) with H2ClB.cntdot.SMe2 gave the primary boranes [2,6-(2-MeC6H4)2C6H3BH2]2, 4, and [2,6-Mes2C6H3BH2]2, 5, resp. of the reaction of 2,6-(4-t-BuC6H4)2C6H3Li with H2ClB.cntdot.SMe2 after 1.5 h with pyridine resulted in the isolation of the primary borane [2,6-(4-t-BuC6H4)2C6H3BH2]2, 2, as the pyridine adduct 2.cntdot.py, which after thermolysis at 190.degree. gave 1-(4-tert-butylphenyl)-7-tert-butyl-9-borafluorene.cntdot.pyridine, 7.cntdot.py. Heating a C6D6 soln. of 4 to 60-70.degree. led to C-H activation and formation of a 1:1 adduct of monomeric 4 and 1-(2-methylphenyl)-5-methyl-9-borafluorene, 12. Reaction of 2 equiv. of 2,6-(4-t-BuC6H4)2C6H3Li with H2ClB.cntdot.SMe2 in hexane soln. followed by addn. of THF gave the very crowded diterphenyl borate [2,6-(4-t-BuC6H4)2C6H3]2B(.mu.-H)2Li[(THF)2]2, 11, which can be converted to 1 by simple addn. of water. Prolonged exposure of 1 to concd. aq. HCl led to B-C bond cleavage and formation of the sterically very crowded diterphenylborinic acid [2,6-(4-t-BuC6H4)2C6H3]2BOH, 15. All compds. have been characterized by 1H, 13C, and 11B NMR spectroscopy and mass spectrometry, and compds. 1, 4, and 11 have also been characterized by single-crystal x-ray crystallog.

RE.CNT 33 THERE ARE 33 CITED REFERENCES AVAILABLE FOR THIS RECORD ALL CITATIONS AVAILABLE IN THE RE FORMAT

L4 ANSWER 13 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

AN 2002:888799 CAPLUS

DN 137:386085

TI Silicone composition polymerizable and/or crosslinkable by cationic process, under thermal activation and by means of a Lewis adduct initiator

IN Sterin, Sebastien; Mignani, Gerard

PA Rhodia Chimie, Fr.

SO PCT Int. Appl., 51 pp.

CODEN: PIXXD2

DT Patent

LA French

Patel

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FAN.CNT 1
       PATENT NO.
                                                                   APPLICATION NO. DATE
                                  KIND DATE
                                           _____
                                  ----
       WO 2002092665
                                                                   WO 2002-FR1617
ΡI
                                   A1
                                           20021121
                                                                                              20020514
             W: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN,
                   CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NO, NZ, OM, PH, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VN, YU, ZA, ZM, ZW, AM, AZ, BY, KG, KZ, MD, RU,
                    TJ, TM
             RW: GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZM, ZW, AT, BE, CH,
                    CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, TR,
                    BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG
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FR 2001-6409 A 20010515 FR 2824835 A1 20021122 FR 2001-6409 20010515

OS MARPAT 137:386085

IT 2118-02-7, Bis (pentafluorophenyl) hydroxyborane

RL: CAT (Catalyst use); USES (Uses)

(silicone compns. crosslinkable by cationically under thermal activation by catalysts based on adducts of Lewis bases with borane Lewis acids)

RN 2118-02-7 CAPLUS

CN Borinic acid, bis(pentafluorophenyl) - (7CI, 8CI, 9CI) (CA INDEX NAME)

AB The crosslinking of silicones is effected cationically at 50-150 degree. by adducts of borane Lewis acids and Lewis bases to give products, useful as release coatings, encapsulants for electronic components, and cladding for optical fibers. A typical coating compn. was prepd. by adding a 2% soln. of (C6F5)3B and 1,4-diazabicyclo[2.2.2]octane in iso-PrOH to 10 g Me3Si(OSiMe2)80(OSiMeR)7OSiMe3 [R = 2-(3,4-epoxycyclohexyl)ethyl] so that the concn. of B in the silicone is 9.5 ppm.

RE.CNT 7 THERE ARE 7 CITED REFERENCES AVAILABLE FOR THIS RECORD
ALL CITATIONS AVAILABLE IN THE RE FORMAT

L4 ANSWER 14 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

AN 2002:863699 CAPLUS

DN 138:304373

TI Transition-metal imido-boroxide complexes: a structural and spectroscopic investigation of the influence of boron

AU Cole, Sarah C.; Coles, Martyn P.; Hitchcock, Peter B.

CS Department of Chemistry, University of Sussex, Falmer, Brighton, BN1 9QJ, UK

SO Journal of the Chemical Society, Dalton Transactions (2002), (22), 4168-4174

CODEN: JCSDAA; ISSN: 1472-7773

PB Royal Society of Chemistry

DT Journal

LA English

IT 20631-84-9

RL: RCT (Reactant); RACT (Reactant or reagent) (coordination; prepn. and structure of molybdenum and titanium imido-boroxide and imido-alkoxide complexes)

RN 20631-84-9 CAPLUS

CN Borinic acid, bis(2,4,6-trimethylphenyl) - (9CI) (CA INDEX NAME)

The four coordinate compds. Mo(NR)2[OB(mes)2]2 (1, R = tBu; 2, R = Ar = 2,6-iPr2C6H3) were prepd. from the reaction of [(mes)2BOLi(Et2O)n]x with the corresponding Mo(NR)2Cl2(dme) starting material; for structural comparison, Mo(NtBu)2[OCH(mes)2]2 (3) was also prepd. The related five-coordinate complexes Ti(NtBu)[OB(mes)2]2(py)2 (4) and Ti(NtBu)[OCH(mes)2]2(py)2 (5) were made using analogous procedures. Crystal structures of 1-5 were investigated to assess the effect of the boron atom on the metal-oxygen and metal-nitrogen(imido) bond lengths and angles. In general, both classes of compd. displayed longer metal-oxygen bonds and shorter metal-nitrogen(imido) bonds for the boroxide derivs. Spectroscopic investigation of the .DELTA. delta. values for the tert-butylimido derivs. revealed that complexes incorporating the [OB(mes)2]- anion exhibit a redn. in the electron d. at the imido nitrogen atom, in agreement with the observations from the solid state structures.

RE.CNT 28 THERE ARE 28 CITED REFERENCES AVAILABLE FOR THIS RECORD ALL CITATIONS AVAILABLE IN THE RE FORMAT

L4 ANSWER 15 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

AN 2002:857650 CAPLUS

DN 138:255269

TI Crystal structure of the strongly hydrogen bonded complex anion [(C6F5)3B(H3O2)B(C6F5)3]-

AU · Drewitt, Mark J.; Niedermann, Martina; Baird, Michael C.

CS Department of Chemistry, Queen's University, Kingston, ON, K7L 3N6, Can.

SO Inorganica Chimica Acta (2002), 340, 207-210 CODEN: ICHAA3; ISSN: 0020-1693

PB Elsevier Science B.V.

DT Journal

LA English

OS CASREACT 138:255269

IT 503026-10-6P

RL: PRP (Properties); SPN (Synthetic preparation); PREP (Preparation) (prepn. and crystal structure of bis[tris(pentafluorophenyl)borane] complex contg. a strongly hydrogen bonded bridging trihydrogen dioxide anion)

RN 503026-10-6 CAPLUS

CN Boron, aquatris(pentafluorophenyl)-, (T-4)-, compd. with dichloromethane and N,N,N',N'-tetramethyl-1,8-naphthalenediamine mono[(T-4)-hydroxytris(pentafluorophenyl)borate(1-)] (1:1:1) (9CI) (CA INDEX NAME)

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10085368.2
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Page 33

CM 1

CRN 155962-45-1 CMF C18 H2 B F15 O CCI CCS

$$F \xrightarrow{G} GH2 \xrightarrow{F} F$$

$$F \xrightarrow{G} F \xrightarrow{F} F$$

$$F \xrightarrow{F} F$$

CM 2

CRN 75-09-2 CMF C H2 Cl2

$C1 - CH_2 - C1$

CM 3

CRN 312640-09-8 CMF C18 H B F15 O . C14 H18 N2 . H

CM 4

CRN 147892-17-9 CMF C18 H B F15 O . H CCI CCS

$$F \longrightarrow C \longrightarrow B \xrightarrow{S+-C} F$$

$$F \longrightarrow F$$

$$F \longrightarrow F$$

$$F \longrightarrow F$$

● H+

CM 5

CRN 20734-58-1 CMF C14 H18 N2

IT 503026-11-7P

RL: PRP (Properties); SPN (Synthetic preparation); PREP (Preparation) (prepn. and mol. structure of bis[tris(pentafluorophenyl)borane] complex contg. a strongly hydrogen bonded bridging trihydrogen dioxide anion)

RN 503026-11-7 CAPLUS

CN Boron, aquatris(pentafluorophenyl)-, (T-4)-, compd. with N,N,N',N'-tetramethyl-1,8-naphthalenediamine mono[(T-4)-hydroxytris(pentafluorophenyl)borate(1-)] (1:1) (9CI) (CA INDEX NAME)

CM 1

CRN 155962-45-1 CMF C18 H2 B F15 O CCI CCS

$$F \xrightarrow{F} GH2 \xrightarrow{F} F$$

$$F \xrightarrow{F} F$$

$$F \xrightarrow{F} F$$

CM 2

CRN 312640-09-8

CMF C18 H B F15 O . C14 H18 N2 . H

CM 3

CRN 147892-17-9

CMF C18 H B F15 O . H

CCI CCS

$$F \longrightarrow C \longrightarrow B \xrightarrow{S+-} F$$

$$F \longrightarrow F$$

$$F \longrightarrow F$$

$$F \longrightarrow F$$

$$F \longrightarrow F$$

●.H+

CM 4

CRN 20734-58-1 CMF C14 H18 N2

Patel

AB The highly electrophilic borane B(C6F5)3 reacts with H2O to form the aqua complex H2OB(C6F5)3.cntdot.2H2O, which is deprotonated by 1,8-bis(dimethylamino)naphthalene (proton sponge) to form [C10H6(NMe2)2H][(C6F5)3B(H3O2)B(C6F5)3]. The complex anion was characterized crystallog. and asymmetry is found, suggesting that this is the 1st example of the theor. predicted asym. H bonded [H3O2] - ion. There also appears to be evidence for significant CF.cntdot..cntdot..cntdot.HO H bonding in the complex anion that may be responsible in part for the overall structure of the anion.

RE.CNT 40 THERE ARE 40 CITED REFERENCES AVAILABLE FOR THIS RECORD ALL CITATIONS AVAILABLE IN THE RE FORMAT

L4 ANSWER 16 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

AN 2002:846231 CAPLUS

DN 138:267844

TI A glucose-selective fluorescence sensor based on boronic acid-diol recognition

AU Karnati, Vishnu Vardhan; Gao, Xingming; Gao, Shouhai; Yang, Wenqian; Ni, Weijuan; Sankar, Sabapathy; Wang, Binghe

CS Department of Chemistry, North Carolina State University, Raleigh, NC, 27695-8204, USA

SO Bioorganic & Medicinal Chemistry Letters (2002), 12(23), 3373-3377 CODEN: BMCLE8; ISSN: 0960-894X

PB Elsevier Science Ltd.

DT Journal

LA English

IT 2622-89-1, Diphenylborinic acid
RL: ARG (Analytical reagent use); DEV (Device component use); ANST (Analytical study); USES (Uses)

(glucose-selective fluorescence sensor based on boronic acid-diol recognition)

RN 2622-89-1 CAPLUS

CN Borinic acid, diphenyl- (6CI, 7CI, 8CI, 9CI) (CA INDEX NAME)



AB A glucose selective diphenylboronic acid fluorescent sensor (10a) with a Ka of 1472 M-1 has been synthesized and evaluated. This sensor shows a 43- and 49-fold selectivity for glucose over fructose and galactose, resp. The binding affinity improvement is about 300-fold and the selectivity improvement for glucose over fructose is about 1400-fold compared with the monoboronic acid compd., phenylboronic acid. 1H NMR studies indicate that sensor 10a binds with .alpha.-d-glucofuranose in a bidentate manner (1:1 ratio).

RE.CNT 28 THERE ARE 28 CITED REFERENCES AVAILABLE FOR THIS RECORD ALL CITATIONS AVAILABLE IN THE RE FORMAT

Patel

L4 ANSWER 17 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

AN 2002:792279 CAPLUS

DN 137:312411

TI Triphenylborons as antifouling agents for fish nets and coatings

IN Yoshimaru, Masaaki; Kohara, Masanori

PA Yoshitomi Fine Chemicals Ltd., Japan

SO Jpn. Kokai Tokkyo Koho, 16 pp.

CODEN: JKXXAF

DT Patent

LA Japanese

FAN.CNT 1

PATENT NO. KIND DATE APPLICATION NO. DATE

----PI JP 2002302494 A2 20021018 JP 2001-402772 20011217
JP 2000-405055 A 20001228

OS MARPAT 137:312411

IT 12113-07-4

RL: RCT (Reactant); RACT (Reactant or reagent)

(triphenylborons as antifouling agents for fishnet and coatings)

RN 12113-07-4 CAPLUS

CN Borate(1-), hydroxytriphenyl-, sodium, (T-4)- (9CI) (CA INDEX NAME)

Na +

GΙ

$$\begin{bmatrix} Ph_{3}B \leftarrow H_{2}N - (Y) - NH_{3}O - C & & & \\ II & & & \\ O & & & D \\ & & & O \end{bmatrix} P \begin{pmatrix} (X) & Q - C - OH_{3}N - (Y) - NH_{2} \rightarrow BPh_{3} \\ & & & \\ O & & & I \\ & & & & \\ O & & & & I \\ \end{bmatrix}$$

$$\begin{bmatrix} Ph_3B \leftarrow N & -Ph_3O - C & -Ph_3N & -Ph_3 &$$

Page 38

AB The compds comprise I (p = q .noteq. 0; if p = 0 and q = 1, then X = C1-18 alkyl, C2-18 alkenyl, aryl, aralkyl, aralkenyl, CHO, etc.; if p = q = 1; then X = C1-18 alkylene, C2-18 alkenylene, arylene, aralkylene, aralkylene, etc.; Y = C2-18 aralkylene; arylene; aralkylene, cycloalkylene) or II (p, q, X = same as above; R1 = halo, C1-18 alkyl; n = 0-1). Thus, Ph3B-NaOH adduct was treated with ethylenediamine at room temp. for 5 h and reacted with oleic acid at room temp. for 2 h to give a compd. Coating contg. 10 wt.% compd. showed no fouling after 6 mo. underwater.

L4 ANSWER 18 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

AN 2002:742334 CAPLUS

DN 138:187447

TI Use of boron enolates in water. The first boron enolate-mediated diastereoselective aldol reactions using catalytic boron sources

AU Mori, Yuichiro; Kobayashi, Juta; Manabe, Kei; Kobayashi, Shu

CS Graduate School of Pharmaceutical Sciences, The University of Tokyo, Bunkyo-ku, Tokyo, 113-0033, Japan

SO Tetrahedron (2002), 58(41), 8263-8268 CODEN: TETRAB; ISSN: 0040-4020

PB Elsevier Science Ltd.

DT Journal

LA English

OS CASREACT 138:187447

IT 2622-89-1 211636-23-6 499774-69-5

RL: CAT (Catalyst use); USES (Uses)

(stereoselective Mukaiyama aldol reactions catalyzed by diarylborinic acids in the presence of surfactant and Bronsted acid in water)

RN 2622-89-1 CAPLUS

CN Borinic acid, diphenyl- (6CI, 7CI, 8CI, 9CI) (CA INDEX NAME)

RN 211636-23-6 CAPLUS

CN Borinic acid, bis[3,5-bis(trifluoromethyl)phenyl] - (9CI) (CA INDEX NAME)

RN 499774-69-5 CAPLUS

CN Boric acid, bis(2,4,6-trimethoxyphenyl) - (9CI) (CA INDEX NAME)

IT 62981-91-3P 66117-64-4P 73774-45-5P 96484-29-6P 176913-70-5P 357437-66-2P 499774-70-8P 499774-71-9P

RL: CAT (Catalyst use); SPN (Synthetic preparation); PREP (Preparation); USES (Uses)

(stereoselective Mukaiyama aldol reactions catalyzed by diarylborinic acids in the presence of surfactant and Bronsted acid in water)

RN 62981-91-3 CAPLUS

CN Borinic acid, di-1-naphthalenyl- (9CI) (CA INDEX NAME)

RN 66117-64-4 CAPLUS

CN Borinic acid, bis(4-methylphenyl) - (9CI) (CA INDEX NAME)

RN 73774-45-5 CAPLUS

CN Borinic acid, bis(4-methoxyphenyl) - (9CI) (CA INDEX NAME)

RN 96484-29-6 CAPLUS

CN Borinic acid, bis(4-bromophenyl) - (9CI) (CA INDEX NAME)

RN 176913-70-5 CAPLUS

CN Borinic acid, bis(4-fluorophenyl)- (9CI) (CA INDEX NAME)

RN 357437-66-2 CAPLUS

CN Borinic acid, bis[4-(trifluoromethyl)phenyl]- (9CI) (CA INDEX NAME)

RN 499774-70-8 CAPLUS

CN Boric acid, di-2-naphthalenyl- (9CI) (CA INDEX NAME)

RN 499774-71-9 CAPLUS

CN Boric acid, bis[3-(trifluoromethyl)phenyl]- (9CI) (CA INDEX NAME)

AB Highly diastereoselective aldol reactions in water using a catalytic amt. of diarylborinic acid have been developed. The reactions proceeded smoothly in the presence of a small amt. of an anionic surfactant and a Bronsted acid. Water was the most suitable solvent; and org. solvents such as ether and dichloromethane were ineffective in this system. Use of bis(4-trifluoromethylphenyl)borinic acid gave high catalytic activity. It is most plausible to conclude that the active species of the reactions are boron enolates, and this is the first example of catalytic use of a boron source in boron enolate-mediated diastereoselective aldol reactions.

RE.CNT 47 THERE ARE 47 CITED REFERENCES AVAILABLE FOR THIS RECORD ALL CITATIONS AVAILABLE IN THE RE FORMAT

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L4 ANSWER 19 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN
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AN 2002:727068 CAPLUS

DN 137:247494

TI Aldol condensation of aromatic aldehydes with silyl enol ethers in water

IN Kobayashi, Osamu; Manabe, Takashi

PA Japan Science and Technology Corporation, Japan

SO Jpn. Kokai Tokkyo Koho, 8 pp.

CODEN: JKXXAF

DT Patent

LA Japanese

FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE			
ΡI	I JP 2002275120		20020925	JP 2001-75091	20010315			
	WO 2002074724	A1	20020926	WO 2002-JP2185	20020308			
	W: US							
	RW: AT, BE,	CH, CY	, DE, DK, ES,	FI, FR, GB, GR, IE	, IT, LU, MC, NL,			
	PT, SE,	TR						

JP 2001-75091 A 20010315

OS MARPAT 137:247494

IT 2622-89-1

RL: CAT (Catalyst use); USES (Uses)

(catalyst; aldol condensation of arom. aldehydes with silyl enol ethers in water)

RN 2622-89-1 CAPLUS

CN Borinic acid, diphenyl- (6CI, 7CI, 8CI, 9CI) (CA INDEX NAME)

Ph | | Ph— B— OH

AB Aldol reaction process comprises reaction of aldehydes with silyl enol ethers in the presence of R1BR2OH [R1, R2 = (un)substituted hydrocarbyl], surfactants, and bronsted acids in water. PhCHO was condensed with MeCH:CPhOSiMe3 in the presence of Ph2BOH, benzoic acid, and sodium dodecyl sulfate in water at 30.degree. for 24 h to give 90% HOCHPhCHMeCOPh with syn/anti 92/8.

L4 ANSWER 20 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

AN 2002:708865 CAPLUS

DN 137:233718

TI Boron-containing silsesquioxanes and their moisture-curable adhesive compositions

IN Miyata, Takeshi; Sugisaki, Toshio

PA Lintec Co., Ltd., Japan

SO Jpn. Kokai Tokkyo Koho, 10 pp.

CODEN: JKXXAF

DT Patent

LA Japanese

FAN.CNT 1

Patel

PATENT NO. KIND DATE APPLICATION NO. DATE
PI JP 2002265609 A2 20020918 JP 2001-71730 20010314

JP 2001-71730 20010314

2622-89-1DP, Diphenylhydroxyborane, reaction products with

silsesquioxane, polymers

RL: IMF (Industrial manufacture); TEM (Technical or engineered material use); PREP (Preparation); USES (Uses)

(boron-contg. silsesquioxanes for weather-resistant moisture-curable adhesives)

2622-89-1 CAPLUS RN

Borinic acid, diphenyl- (6CI, 7CI, 8CI, 9CI) (CA INDEX NAME) CN

AB The invention relates to silsesquioxanes having B liking to Si via O. Thus, PhSi(OMe)3 and Et2BOMe were reacted in the presence of methanesulfonic acid to give a B-terminated silsesquioxane, which was applied on a plastic film and cured to show good weather resistance.

ANSWER 21 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

2002:671913 CAPLUS AN

DN 137:201430

ΤI Process for preparing aromatic boronic and borinic acids

IN Meudt, Andreas; Erbes, Michael; Forstinger, Klaus

PA Clariant G.m.b.H., Germany

Eur. Pat. Appl., 9 pp. SO

CODEN: EPXXDW

DT Patent

LΑ German

FAN.CNT 1

	-				
PA	TENT NO.	KIND DATE	AP	PLICATION NO.	DATE
PI EP	1236730	A2 2002	0904 EP	2002-3691	20020219
	R: AT, BE,	CH, DE, DK,	ES, FR, GB,	GR, IT, LI, LU	, NL, SE, MC, PT,
			RO, MK, CY,	· · · · · · · · · · · · · · · · · · ·	, , , , , , , , , , , , , , , , , , , ,
			DE	2001-10110051	A 20010302
DE	10110051	A1 2002	0912 DE	2001-10110051	20010302
DE	10110051	C2. 2003	0703		
US	2002161230	A1 2002	1031 US	2002-85368	20020228
			DE	2001-101100512	A 20010302
JP	2002308883	A2 2002	1023 JP	2002-57278	20020304
			DE	2001-101100512	A 20010302

OS CASREACT 137:201430; MARPAT 137:201430

IT 66117-64-4P, Di(p-tolyl)borinic acid

RL: SPN (Synthetic preparation); PREP (Preparation) (prepn. of)

RN 66117-64-4 CAPLUS

CNBorinic acid, bis(4-methylphenyl) - (9CI) (CA INDEX NAME)

GΙ

Ι

ΙI

AB The process for prepn. of title compds., I and II (R1-R5 = H, Me, straight or branched C1-8 alkyl, F, CnH2n+1-f, n = 1-8, f = 1-2n+1 F, CH(OC1-5 alkyl)2, C(C1-5 alkyl), (OC1-5 alkyl), CH2(OC1-5 alkyl), CHMe(OC1-5 alkyl), C1-5 alkoxy, N(C1-5 alkyl)2, (un)substituted Ph, X, Y, Z = (un)substituted O, N, etc.), by the reaction of chloroarom. with BW'W''W''' (W', W''', W''' = C1-6 alkoxy, F, Cl, Br, I, diorganoamino, organothio, etc.), in a solvent at temp. of -100.degree. to 80.degree. is described. Thus, reaction of PhCl with Li in THF followed by treatment with B(OEt)3 and acidic hydrolysis gave 97% phenylboronic acid.

L4 ANSWER 22 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

AN 2002:643255 CAPLUS

DN 138:296525

TI (I) Zinc aqua and alkoxide complexes: models of zinc enzymes. (II) estimation of the Bronsted acidity of aquatris(perfluorophenyl)borane

AU Bergquist, Catherine Jones

CS Columbia Univ., New York, NY, USA

SO (2001) 165 pp. Avail.: UMI, Order No. DA3028501 From: Diss. Abstr. Int., B 2002, 62(10), 4534

DT Dissertation

LA English

IT 155962-45-1

RL: PRP (Properties)
(Bronsted acidity of)

RN 155962-45-1 CAPLUS

CN Boron, aquatris(pentafluorophenyl)-, (T-4)- (9CI) (CA INDEX NAME)

$$F \longrightarrow F \longrightarrow F \longrightarrow F \longrightarrow F$$

AB Unavailable

L4 ANSWER 23 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

AN 2002:567539 CAPLUS

DN 137:278620

TI Surface-catalyzed degradation of tetraphenylborate on mineral and oxide surfaces

AU Mills, Gary L.; Sandhu, Shingara S.

CS Savannah River Ecology Laboratory, University of Georgia, Aiken, SC, 29802, USA

SO Preprints of Extended Abstracts presented at the ACS National Meeting, American Chemical Society, Division of Environmental Chemistry (2002), 42(2), 152-155

CODEN: PEACF2; ISSN: 1524-6434

PB American Chemical Society, Division of Environmental Chemistry

DT Journal; (computer optical disk)

LA English

IT 2622-89-1, Diphenylborinic acid

RL: FMU (Formation, unclassified); FORM (Formation, nonpreparative) (formation in surface-catalyzed degrdn. of tetraphenylborate on mineral and oxide surfaces)

RN 2622-89-1 CAPLUS

CN Borinic acid, diphenyl- (6CI, 7CI, 8CI, 9CI) (CA INDEX NAME)

The rates of surface-catalyzed degrdn. of tetraphenylborate on Fe2O3, TiO2, Al2O3, and MnO2 were compared. The effect of dioxygen and diphenylboric acid (DPBA) on the TPB degrdn. rate was examd. Kaolinite and montmorillonite were obtained from the ref. mineral collection at the Department of Geol., University of Missouri. The rates of surface-catalyzed degrdn. followed the order TiO2 = Fe2O3 > kaolinite = montmorillonite > MnO2 >> Al2O3. The reaction appeared to be biphasic for TiO2, MnO2, and Fe2O3 with an initial fast reaction followed by a slower degrdn. rate. The primary degrdn. products detected were DPBA and biphenyl. Degrdn. rates were faster on the Ca-satd. kaolinite and montmorillonite clay surfaces compared to the Na-satd. clays. The degrdn. rates on untreated clays were similar to that obsd. for the Ca-satd. minerals. Oxygen was required to maintain TPB oxidn. after the initial Lewis acid sites were reduced.

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10085368.2
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Page 45

RE.CNT 6 THERE ARE 6 CITED REFERENCES AVAILABLE FOR THIS RECORD ALL CITATIONS AVAILABLE IN THE RE FORMAT

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ANSWER 24 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN
T.4
AN
     2002:555442 CAPLUS
DN
     137:110887
     Process for the narrow-range alkoxylation of fatty alcohols using a
ΤI
     substituted phenylboron-containing catalyst
IN
     Priou, Christian B.; Beurdeley, Patricia
     Rhodia, Inc., USA
PA
     PCT Int. Appl., 18 pp.
SO
     CODEN: PIXXD2
DT
     Patent
     English
LA
FAN.CNT 1
     PATENT NO.
                       KIND DATE
                                              APPLICATION NO.
                       _ _ _ _
PΙ
     WO 2002057209
                        A1
                              20020725
                                              WO 2002-US1024
                                                                20020110
         W: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN,
              CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, ES, FI, GB, GD, GE, GH,
              GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR,
              LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NO, NZ, PL, PT,
              RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, TZ, UA, UG, UZ,
              VN, YU, ZA, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM
         RW: GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZM, ZW, AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, TR,
              BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG
                                              US 2001-766533 A 20010119
                                              US 2001-766533
     US 2002128521
                        Α1
                              20020912
                                                               20010119
     US 6593500
                        B2
                              20030715
OS
     MARPAT 137:110887
     147892-17-9 155962-46-2
     RL: CAT (Catalyst use); USES (Uses)
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IT

(process for the narrow-range alkoxylation of fatty alcs. using a substituted phenylboron-contg. catalyst)

RN 147892-17-9 CAPLUS

CNBorate(1-), hydroxytris(pentafluorophenyl)-, hydrogen, (T-4)- (9CI) (CA INDEX NAME)

$$F \longrightarrow C^{-} \xrightarrow{B} \xrightarrow{3+} C \longrightarrow F$$

$$F \longrightarrow F \longrightarrow F$$

$$F \longrightarrow F$$

$$F \longrightarrow F$$

RN 155962-46-2 CAPLUS

CN Boron, (methanol)tris(pentafluorophenyl)-, (T-4)- (9CI) (CA INDEX NAME)

AB Narrow-range alkoxylates of org. compds. are prepd. by: (a) providing an active hydrogen org. compd. (e.g., 1-dodecanol) having 1-22 carbon atoms; and (b) alkoxylating the org. compd. with an alkylene oxide (e.g., ethylene oxide) in the presence of a phenylboron-contg. catalyst BX3 [e.g, tris(pentafluorophenyl)borane] or HBX4 (X = a Ph moiety having substituents selected from 1-5 fluorine atoms, 1-5 CF3 moieties, 1-5 OCF3 or SCF3 moieties, OR; R = H, alkyl or aryl group having 1-22 carbon atoms). The process affords an alkoxylate product having a very narrow mol.-wt. distribution with a low degree of both residual org. starting material and polyoxyalkylenes.

RE.CNT 3 THERE ARE 3 CITED REFERENCES AVAILABLE FOR THIS RECORD ALL CITATIONS AVAILABLE IN THE RE FORMAT

L4 ANSWER 25 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

AN 2002:466016 CAPLUS

DN 137:33411

TI Method for purifying fluoroarylboron derivative and bis(fluoroaryl)boron derivative

IN Ikeno, Ikuyo; Mitsui, Hitoshi; Iida, Toshiya; Moriguchi, Toshimitsu

PA Nippon Shokubai Co., Ltd., Japan

SO PCT Int. Appl., 71 pp. CODEN: PIXXD2

DT Patent

LA Japanese

FAN.CNT 1

APPLICATION NO. PATENT NO. KIND DATE _ _ _ _ -----PΙ WO 2002048156 WO 2001-JP10791 Α1 20020620 20011210 W: CN, IL, JP, US RW: AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, TR JP 2000-376612 A 20001211 EP 1342725 A1 20030910 EP 2001-270538 20011210 R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, FI, CY, TR JP 2000-376612 A 20001211 WO 2001-JP10791W 20011210 US 2003050282 Α1 20030313 US 2002-220635 20020904 JP 2000-376612 A 20001211 WO 2001-JP10791W 20011210

Patel

OS MARPAT 137:33411

IT 2118-02-7P, Bis (pentafluorophenyl) borinic acid

RL: IMF (Industrial manufacture); PUR (Purification or recovery); SPN (Synthetic preparation); PREP (Preparation)

(method for purifying fluoroarylboron deriv. and bis(fluoroaryl)boron
deriv.)

RN 2118-02-7 CAPLUS

CN Borinic acid, bis(pentafluorophenyl) - (7CI, 8CI, 9CI) (CA INDEX NAME)

AB A soln. comprising a fluoroarylboron deriv., a bis(fluoroaryl)boron deriv., and a hydrocarbon solvent is treated to ppt. the fluoroarylboron deriv., which is isolated by first filtration. The resultant filtrate is cooled to ppt. the bis(fluoroaryl)boron deriv., which is isolated by second filtration. In the case where the soln. contains a fluorobenzene, the fluorobenzene is removed by concg. the soln. Thus, a fluoroarylboron deriv. and bis(fluoroaryl)boron deriv. which are free from impurities and have high purity can be easily produced at low cost.

Bis(pentafluorophenyl)borinic acid with 100% purity was obtained using the title method.

RE.CNT 3 THERE ARE 3 CITED REFERENCES AVAILABLE FOR THIS RECORD ALL CITATIONS AVAILABLE IN THE RE FORMAT

L4 ANSWER 26 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

AN ·2002:444647 CAPLUS

DN 137:263332

TI A study of representative alcohol, alkoxide, thiol and thiolate complexes of B(C6F5)3; their roles as activators of zirconocene olefin polymerization initiators

AU Drewitt, Mark J.; Niedermann, Martina; Kumar, Rajesh; Baird, Michael C.

CS Department of Chemistry, Queen's University, Kingston, ON, K7L 3N6, Can.

SO Inorganica Chimica Acta (2002), 335, 43-51 CODEN: ICHAA3; ISSN: 0020-1693

PB Elsevier Science B.V.

DT Journal

LA English

IT 155962-39-3P

RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)

(alc., alkoxide, thiol and thiolate complexes of B(C6F5)3 as activators for zirconocene olefin polymn. initiators)

RN 155962-39-3 CAPLUS

CN Boron, (1-octadecanol)tris(pentafluorophenyl)-, (T-4)- (9CI) (CA INDEX NAME)

ΙŢ

The highly electrophilic borane B(C6F5)3 reacts with n-octadecanol (n-C18H37OH) and n-octadecanethiol (n-C18H37SH) to form equil. mixts. of reactants and the 1:1 adducts (n-C18H37EH)B(C6F5)3 (E=O, S); equil. consts. for adduct formation are detd. The adducts are deprotonated by 1,8-bis(dimethylamino)naphthalene (proton sponge) to form the salts [C10H6(NMe2)2H][(n-C18H37O)B(C6F5)3] and [C10H6(NMe2)2H][(n-C18H37S)B(C6F5)3], resp., and by Cp2ZrMe2 to give methane and, apparently, the unstable zirconium complexes [Cp2ZrMe][(n-C18H37E)B(C6F5)3]. The alc., alkoxide, thiol and thiolate systems are characterized in soln. by 1H, 19F, 13C{1H} and 11B NMR spectroscopy and in the solid state by FAB mass spectrometry, and it is also shown that proton sponge can coordinate in monodentate fashion to B(C6F5)3.

RE.CNT 57 THERE ARE 57 CITED REFERENCES AVAILABLE FOR THIS RECORD ALL CITATIONS AVAILABLE IN THE RE FORMAT

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ANSWER 27 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN
L4
ΑN
     2002:428913 CAPLUS
DN
     137:6275
ΤI
     Process for preparing bis(fluoroaryl)boron derivatives
     Ikeno, Ikuyo; Mitsui, Hitoshi; Iida, Toshiya; Moriguchi, Toshimitsu
ΤN
PA
     Nippon Shokubai Co., Ltd., Japan
SO
     PCT Int. Appl., 29 pp.
     CODEN: PIXXD2
DT
     Patent
T<sub>1</sub>A
     Japanese
FAN.CNT 1
     PATENT NO.
                      KIND DATE
                                            APPLICATION NO.
                                                             DATE
                      ____
PΙ
     WO 2002044185
                       A1
                            20020606
                                            WO 2001-JP10392
                                                             20011128
         W: CN, IL, JP, US
         RW: AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL,
             PT, SE, TR
                                            JP 2000-369621 A 20001130
     EP 1338601
                            20030827
                                            EP 2001-998551
                       Α1
                                                            20011128
         R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT,
             IE, FI, CY, TR
                                            JP 2000-369621 A 20001130
                                            WO 2001-JP10392W 20011128
     US 2003045507
                       Α1
                            20030306
                                            US 2002-221029
                                                            20020909
                                            JP 2000-369621 A 20001130
                                            WO 2001-JP10392W 20011128
     CASREACT 137:6275; MARPAT 137:6275
OS
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Patel 9/24/2003>

2118-02-7P, Bis (pentafluorophenyl) borinic acid

RL: IMF (Industrial manufacture); SPN (Synthetic preparation); PREP (Preparation)

(process for prepg. bis(fluoroaryl)boron derivs.)

RN 2118-02-7 CAPLUS

CN Borinic acid, bis(pentafluorophenyl) - (7CI, 8CI, 9CI) (CA INDEX NAME)

AB A bis(fluoroaryl)boron deriv. is prepd. by reacting a tris(fluoroaryl)boron with water or the like at a molar ratio ranging from 1:0.9 to 1:1.1 in a hydrocarbon solvent. The title compds. are useful as intermediates for pharmaceuticals, agrochems., polymn. catalysts, etc. It is preferable that the reaction be carried out while the solvent is distd. off and the solvent consist substantially of an aliph. hydrocarbon. Thus, high-purity bis(fluoroaryl)boron derivs. can be prepd. and isolated easily and inexpensively. Thus, a mixt. of tris(pentafluorophenyl)boron and water in Isopar E was heated at 100.degree. for 4 h to give bis(pentafluorophenyl)borinic acid in 99% yield, vs. 41% yield in a ref. process.

RE.CNT 2 THERE ARE 2 CITED REFERENCES AVAILABLE FOR THIS RECORD ALL CITATIONS AVAILABLE IN THE RE FORMAT

L4 ANSWER 28 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

AN 2002:428912 CAPLUS

DN 137:20467

TI Preparation of diarylborinic acid esters as DNA methyl transferase inhibitors

IN Benkovic, Stephen J.; Shapiro, Lucille; Baker, Stephen J.; Wahnon, Daphne
C.; Wall, Mark; Shier, Vincent K.; Scott, Charles P.; Baboval, Justin

PA The Penn State Research Foundation, USA

SO PCT Int. Appl., 35 pp. CODEN: PIXXD2

DT Patent

LA English

FAN.CNT 1

	PATENT NO.				ND :	D DATE			APPLICATION NO.					DATE				
PI	WO 2002044184 WO 2002044184					20020606 20030227		WO 2001-US45129				29	20011129					
				A.	3													
	W :	ΑE,	AG,	AL,	AM,	AT,	AU,	ΑŻ,	BA,	BB,	BG,	BR,	BY,	ΒZ,	CA,	CH,	CN,	
		CO,	CR,	CU,	CZ,	DE,	DK,	DM,	DZ,	EE,	ES,	FI,	GB,	GD,	GE,	GH,	GM,	
		HR,	HU,	ID,	IL,	IN,	IS,	JP,	KE,	KG,	KP,	KR,	ΚZ,	LC,	LK,	LR,	LS,	
		LT,	LU,	LV,	MA,	MD,	MG,	MK,	MN,	MW,	MX,	MZ,	NO,	NZ,	ΡL,	PT,	RO,	
		RU,	SD,	SE,	SG,	SI,	SK,	SL,	ΤJ,	TM,	TR,	TT,	TZ,	UΑ,	UG,	US,	UZ,	
		VN,	YU,	ZA,	ZW,	AM,	ΑZ,	BY,	KG,	ΚZ,	MD,	RU,	TJ,	TM				
	RW:	GH,	GM,	KΕ,	LS,	MW,	MZ,	SD,	SL,	SZ,	TZ,	UG,	ZM,	ZW,	AT,	BE,	CH,	
		CY,	DE,	DK,	ES,	FI,	FR,	GB,	GR,	ΙE,	IT,	LU,	MC,	NL,	PΤ,	SE,	TR,	
		BF,	ВJ,	CF,	CG,	CI,	CM,	GA,	GN,	GQ,	GW,	ML,	MR,	ΝE,	SN,	TD,	TG	
		US 2000-250202PP 20001130																

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10085368.2
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Page 50

AU 2002039407 **A5** 20020611 AU 2002-39407 US 2000-250202PP 20001130 WO 2001-US45129W 20011129 EP 1339725 20030903 A2 EP 2001-987166 20011129 R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, SI, LT, LV, FI, RO, MK, CY, AL, TR US 2000-250202PP 20001130 WO 2001-US45129W 20011129 CASREACT 137:20467; MARPAT 137:20467 IT 2622-89-1P, Diphenylborinic acid 73774-45-5P, Di(4-methoxyphenyl)borinic acid 89566-59-6P, Di (4-chlorophenyl) borinic acid 161800-66-4P, Di(3,4-methylenedioxyphenyl)borinic acid 176913-70-5P, Di(4-fluorophenyl)borinic acid 433338-06-8P, Di (3-chlorophenyl) borinic acid 433338-07-9P, Di(4-chloro-2-fluorophenyl)borinic acid RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent) (prepn. and esterification of) RN2622-89-1 CAPLUS Borinic acid, diphenyl- (6CI, 7CI, 8CI, 9CI) (CA INDEX NAME) CN

RN 73774-45-5 CAPLUS CN Borinic acid, bis(4-methoxyphenyl)- (9CI) (CA INDEX NAME)

RN 89566-59-6 CAPLUS CN Borinic acid, bis(4-chlorophenyl)- (9CI) (CA INDEX NAME)

RN 161800-66-4 CAPLUS CN Borinic acid, bis(1,3-benzodioxol-5-yl)- (9CI) (CA INDEX NAME)

RN 176913-70-5 CAPLUS

CN Borinic acid, bis(4-fluorophenyl) - (9CI) (CA INDEX NAME)

RN 433338-06-8 CAPLUS

CN Boronic acid, bis(3-chlorophenyl) - (9CI) (CA INDEX NAME)

RN 433338-07-9 CAPLUS

CN Boronic acid, bis(4-chloro-2-fluorophenyl) - (9CI) (CA INDEX NAME)

AB This invention provides prepn. of diarylborinic acid esters as broad-spectrum antibiotics that are inhibitors of bacterial adenine DNA methyltransferases. Thus, reaction of dichloroborane di-Me sulfide complex with 4-FC6H4MgBr in THF followed by acidic workup gave di(4-fluorophenyl)borinic acid which on treatment with 8-hydroxyquinoline in EtOH gave di(4-fluorophenyl)borinic acid 8-hydroxyquinoline ester. The biol. activity of the compds. prepd. are given.

- L4 ANSWER 29 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN
- AN 2002:296406 CAPLUS
- DN 137:47258
- TI Surface organometallic chemistry of main group elements: selective synthesis of silica supported [.tplbond.Si-OB(C6F5)3]-[HNEt2Ph]+
- AU Millot, Nicolas; Cox, Andrew; Santini, Catherine C.; Molard, Yann; Basset, Jean-Marie
- CS Laboratoire de Chimie Organometallique de Surface, CNRS-ESCPE Lyon, Villeurbanne, 69626, Fr.
- SO Chemistry--A European Journal (2002), 8(6), 1438-1442 CODEN: CEUJED; ISSN: 0947-6539

PB Wiley-VCH Verlag GmbH Journal DT English LΑ CASREACT 137:47258 OS ΙT 438536-83-5P

RL: PRP (Properties); SPN (Synthetic preparation); PREP (Preparation) (prepn. and modeling of partially dehydroxylated silica-supported ammonium borate by)

RN438536-83-5 CAPLUS

Borate(1-), hydroxytris(pentafluorophenyl)-, (T-4)-, hydrogen, compd. with CN N, N-diethylbenzenamine (1:1) (9CI) (CA INDEX NAME)

CM 1

CRN 147892-17-9 CMF C18 H B F15 O . H CCI CCS

$$F \xrightarrow{F} F \xrightarrow{OH^{-}} F \xrightarrow{F} F$$

$$F \xrightarrow{F} F \xrightarrow{F} F$$

Н+

CM2

CRN 91-66-7 CMF C10 H15 N

Ph Et-N-Et

438536-83-5DP, partially dehydroxylated silica-supported ΙT with/without deuterium and partial oxygen-18 labeling of silica RL: PRP (Properties); SPN (Synthetic preparation); PREP (Preparation) (selective synthesis and mol. modeling of)

RN 438536-83-5 CAPLUS

Borate(1-), hydroxytris(pentafluorophenyl)-, (T-4)-, hydrogen, compd. with CN N, N-diethylbenzenamine (1:1) (9CI) (CA INDEX NAME)

CM 1

Patel

CRN 147892-17-9 CMF C18 H B F15 O . H CCI CCS

$$F = \begin{bmatrix} C & OH & F & F \\ & 3+ & C & F \\ & & F & F \end{bmatrix}$$

● H+

CM 2

CRN 91-66-7 CMF C10 H15 N

The reaction of the Lewis acid B(C6F5)3 with silanol groups of SiO2 surfaces, dehydroxylated at different temps. (300, 500, 700, and 800.degree.), was studied in presence of the Bronsted base NEt2Ph. The structure of the resulting modified SiO2 supports [.tplbond.Si-OB(C6F5)3]-[HNEt2Ph]+ (1) was carefully identified by IR and multinuclear solid-state NMR spectroscopies, isotopic 2H and 180 labeling, elemental anal., mol. modeling, and comparison with synthesized mol. models. Highly dehydroxylated SiO2 surfaces were required to transform selectively each silanol group into unique [.tplbond.Si-OB(C6F5)3]-[HNEt2Ph]+ fragments. For lower dehydroxylation temps., two sorts of surface sites were coexisting on SiO2: the free silanol groups [.tplbond.SiOH] and the ionic species 1.

RE.CNT 60 THERE ARE 60 CITED REFERENCES AVAILABLE FOR THIS RECORD ALL CITATIONS AVAILABLE IN THE RE FORMAT

L4 ANSWER 30 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

AN 2002:252591 CAPLUS

DN 137:6242

TI Reactions of Hydroxymesitylboranes with Metal Alkyls: An Approach to New Sterically Hindered (Metaloxy)mesitylboranes

AU Anulewicz-Ostrowska, Romana; Lulinski, Sergiusz; Pindelska, Edyta; Serwatowski, Janusz

CS Faculty of Chemistry, University of Warsaw, Warsaw, 02-093, Pol. SO Inorganic Chemistry (2002), 41(9), 2525-2528 CODEN: INOCAJ; ISSN: 0020-1669 PB American Chemical Society DT Journal LA . English OS CASREACT 137:6242 ΙT 20631-84-9 (prepn. of) RN 20631-84-9 CAPLUS CN Borinic acid, bis(2,4,6-trimethylphenyl) - (9CI) (CA INDEX NAME)

AB Reactions of mesitylboronic acid with alkyl derivs. of aluminum R3Al (R = Me, Et, Bui), gallium (Me3Ga), and zinc (Et2Zn) were investigated. The treatment of mesitylboronic acid, MesB(OH)2, with trimethylgallium afforded the discrete dimer [.mu.-(MesB(OH)O)GaMe2]2 (1), which is the simple example of a O-metalated boronic acid with no hydrogen bonding in the crystal lattice. In addn., the reaction of dimesitylborinic acid, Mes2BOH, with diethylzinc produced the low-valent zinc compd. [(.mu.-Mes2BO)ZnEt]2 (2), which was also characterized by x-ray diffraction.

RE.CNT 19 THERE ARE 19 CITED REFERENCES AVAILABLE FOR THIS RECORD ALL CITATIONS AVAILABLE IN THE RE FORMAT

L4 ANSWER 31 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

AN 2002:67407 CAPLUS

DN 136:288131

TI Reactivity of the B-H Bond in Tris(pyrazolyl)hydroborato Zinc Complexes:
Unexpected Example of Zinc Hydride Formation in a Protic Solvent and Its
Relevance towards Hydrogen Transfer to NAD+ Mimics by
Tris(pyrazolyl)hydroborato Zinc Complexes in Alcoholic Media

AU Bergquist, Catherine; Koutcher, Lawrence; Vaught, Amanda L.; Parkin, Gerard

CS Department of Chemistry, Columbia University, New York, NY, 10027, USA

SO Inorganic Chemistry (2002), 41(4), 625-627 CODEN: INOCAJ; ISSN: 0020-1669

PB American Chemical Society

DT Journal

LA English

IT 405879-87-0

RL: RCT (Reactant); RACT (Reactant or reagent) (attempted hydrogen transfer reaction from)

RN 405879-87-0 CAPLUS

CN Zinc(1+), aqua-d2-[tris[3-(1,1-dimethylethyl)-5-methyl-1H-pyrazolato-.kappa.N1]hydroborato(1-)-.kappa.N2,.kappa.N2',.kappa.N2'']-, (T-4)-, (T-4)-hydroxy-d-tris(pentafluorophenyl)borate(1-) (9CI) (CA INDEX NAME)

CM 1

CRN 405879-86-9 CMF C18 B D F15 O CCI CCS

CM 2

CRN 405879-85-8 CMF C24 H40 B D2 N6 O Zn CCI CCS

Me N N Bu-t
$$Zn^{2+}$$
 O D Me N Bu-t D

IT 243121-77-9

RL: PEP (Physical, engineering or chemical process); PYP (Physical process); PROC (Process) (dissoln. in deuterated methanol)

RN 243121-77-9 CAPLUS

CN Zinc(1+), aqua[tris[3-(1,1-dimethylethyl)-5-methyl-1H-pyrazolato-.kappa.N1]hydroborato(1-)-.kappa.N2',.kappa.N2',.kappa.N2'']-, (T-4)-hydroxytris(pentafluorophenyl)borate(1-) (9CI) (CA INDEX NAME)

CM 1

CRN 243121-76-8 CMF C24 H42 B N6 O Zn CCI CCS

Me N N N
$$= N^{-1}$$
 N $= N^{-1}$ N $= N^{-1$

CM 2

CRN 148657-98-1 CMF C18 H B F15 O CCI CCS

IT 405879-95-0

RL: RCT (Reactant); RACT (Reactant or reagent) (zinc deuteride complex from dissoln. of)

RN 405879-95-0 CAPLUS

CN Zinc(1+), aqua[tris[3-(1,1-dimethylethyl)-5-methyl-1H-pyrazolato-.kappa.N1]hydro-d-borato(1-)-.kappa.N2,.kappa.N2',.kappa.N2'']-, (T-4)-, (T-4)-hydroxytris(pentafluorophenyl)borate(1-) (9CI) (CA INDEX NAME)

CM 1

CRN 405879-94-9

CMF C24 H41 B D N6 O Zn

CCI . CCS

Me N N N
$$2n^{2+}$$
 OH2

Me N Bu-t

Me Bu-t

CM 2

CRN 148657-98-1 CMF C18 H B F15 O CCI CCS

AB Solns. of the Zn hydroxide complex [TpBut,Me] ZnOH in alcs. (ROH; R = Me, Et, Pri) achieve hydride transfer to the NAD+ model, 10-methylacridinium perchlorate. D labeling studies, however, demonstrate that the source of the hydride is not the alc. but, rather, the B-H group of the [TpBut,Me] ligand. A further example in which a [TpBut,Me] ligand acts as a hydride donor is provided by the reaction of the aqua complex {[TpBut,Me]Zn(OH2)}[HOB(C6F5)3] with MeOH to generate the Zn hydride complex [TpBut,Me]ZnH. The present study therefore provides a caveat for the often assumed inertness of the B-H group in tris(pyrazolyl)hydroborato ligands, esp. in the presence of reactive cationic species.

RE.CNT 37 THERE ARE 37 CITED REFERENCES AVAILABLE FOR THIS RECORD ALL CITATIONS AVAILABLE IN THE RE FORMAT

L4 ANSWER 32 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

AN 2002:45655 CAPLUS

DN 137:33348

TI A new organodithiophosphoric derivative; synthesis and structural characterization of bis(diphenylborano)dithiophosphoric [(C6H5)2BO]2P(S)SH

AU Gabriela, Cretiu; Reka, Torok; Delia, Bugnariu; Oxana, Jeman;

Silaghi-Dumitrescu, Ioan
CS Universitatea "Babes-Bolvai" Fac

CS Universitatea "Babes-Bolyai", Facultatea de Chimie si Inginerie Chimica, Cluj-Napoca, RO-3400, Rom.

SO Studia Universitatis Babes-Bolyai, Chemia (1999), 44(1-2), 177-182

Patel

10085368.2 Page 58

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CODEN: SUBCAB; ISSN: 1224-7154
PB
     Studia Universitatis Babes-Bolyai
DT
     Journal
     English
LΑ
     CASREACT 137:33348
OS
IT
     2622-89-1P
     RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT
      (Reactant or reagent)
         (prepn. and reaction with phosphorus pentasulfide to give
         bis (diphenylborano) dithiophosphoric)
RN
     2622-89-1 CAPLUS
     Borinic acid, diphenyl- (6CI, 7CI, 8CI, 9CI) (CA INDEX NAME)
CN
   Ph
Ph-B-OH
AB
     A new organo deriv. of dithiophosphoric acid (RO) 2P(S) SH was obtained by
     the reaction of P pentasulfide (P4S10) with diphenylborinic acid. IR, 1H,
     13C, 11B and 31P NMR spectra of intermediates and the main product are
     discussed. Geometrical parameters (distances in , angles in degrees) for
     the min. energy structure were studied by ab initio RHF/3-21G* using
     Spartan version 5.0 installed on a SGI Octane.
                THERE ARE 12 CITED REFERENCES AVAILABLE FOR THIS RECORD
RE.CNT 12
                ALL CITATIONS AVAILABLE IN THE RE FORMAT
     ANSWER 33 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN
L4
     2002:10467 CAPLUS
AN
     136:69823
DN -
     Preparation of imidazole derivatives or salts thereof and drugs containing
ΤI
     the derivatives or the salts
IN
     Konno, Fujiko; Nagao, Yoshihiro; Isomae, Kazuo; Ohtsuka, Mari; Takahashi,
     Yoshiyuki; Ishii, Fumio; Hirota, Hiroyuki; Takeda, Sunao; Kawamoto,
     Noriyuki; Honda, Haruyoshi; Sato, Susumu
     Ssp Co., Ltd., Japan
PA
     PCT Int. Appl., 53 pp.
SO
     CODEN: PIXXD2
DT
     Patent
     Japanese
T.A
FAN.CNT 1
     PATENT NO.
                        KIND DATE
                                                APPLICATION NO. DATE
                        ____
                                                -----
                                                WO 2001-JP4836
PΙ
     WO 2002000648
                         A1
                               20020103
                                                                  20010608
          W: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN,
              CR, CU, CZ, DE, DK, DM, DZ, EE, ES, FI, GB, GD, GE, GH, GM, HR,
              HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT,
          LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, TZ, UA, UG, US, UZ, VN, YU, ZA, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM

RW: GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZW, AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, TR, BF,
               BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG
                                                JP 2000-194024 A 20000628
     AU 2001064223
                                                AU 2001-64223
                          Α5
                               20020108
                                                                   20010608
                                                JP 2000-194024 A 20000628
```

WO 2001-JP4836 W 20010608

10085368.2

Page 59

EP 1295880 A1 20030326 EP 2001-938563 20010608
R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, SI, LT, LV, FI, RO, MK, CY, AL, TR

JP 2000-194024 A 20000628

JP 2000-194024 A 20000628 WO 2001-JP4836 W 20010608

OS MARPAT 136:69823

IT 385414-95-9

RL: RCT (Reactant); RACT (Reactant or reagent)
 (prepn. of imidazole derivs. or salts thereof and drugs contg. derivs.
 or salts)

RN 385414-95-9 CAPLUS

CN Borinic acid, (2,4-difluorophenyl)phenyl- (9CI) (CA INDEX NAME)

GΙ

$$\begin{array}{c|c}
R^1 & X^1 & Y - (CH_2)_{m} - Z \\
R^2 & X^2 & R^3
\end{array}$$

$$\begin{array}{c|c}
X^1 & Y - (CH_2)_{m} - Z \\
R & R^4
\end{array}$$

$$\begin{array}{c|c}
X^1 & Y - (CH_2)_{m} - Z \\
R & R^4
\end{array}$$

Ph N O-CH₂CH₂-O-CH₂-N N

AB Title compds. [I; R1, R2 each independently = aryl, heteroaryl; A, X1, X2 each independently = N, CH; Y, Z each independently = O, S, NH, SO2, CH2, NCH3; R3, R4, R5 each independently = H, alkyl, NH2, alkoxy, Cl; m = 1, 2, 3, 4; n = 0, 1, 2, 3, 4] and salts are prepd. and formulation discussed. Title compds. I exhibit excellent inhibitory activities against the prodn. of NO and IL-6 and are useful in the prevention or treatment of diseases resulting from over-development of NO and IL-6. Thus, the title compd. II was prepd. and tested as antiinflammatory in male ICR mouse with inhibition result at 30.5% for 3 mg/kg dosage.

Ι

ΙI

RE.CNT 6 THERE ARE 6 CITED REFERENCES AVAILABLE FOR THIS RECORD

ALL CITATIONS AVAILABLE IN THE RE FORMAT

- L4 ANSWER 34 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN
- AN 2001:918130 CAPLUS
- DN 136:183894
- TI Anion Stability in Stannylium, Oxonium, and Silylium Salts of the Weakly Coordinating Anion [C6F4-1,2-{B(C6F5)2}2(.mu.-OCH3)]-
- AU Henderson, Lee D.; Piers, Warren E.; Irvine, Geoffry J.; McDonald, Robert
- CS Department of Chemistry, University of Calgary, Calgary, AB, T2N 1N4, Can.
- SO Organometallics (2002), 21(2), 340-345 CODEN: ORGND7; ISSN: 0276-7333
- PB American Chemical Society
- DT Journal
- LA English
- OS CASREACT 136:183894
- IT 400644-12-4P 400644-13-5P

RL: SPN (Synthetic preparation); PREP (Preparation) (prepn. of)

- RN 400644-12-4 CAPLUS
- CN Boron, [2-[bis(pentafluorophenyl)boryl]-3,4,5,6tetrafluorophenyl] (methanol)bis(pentafluorophenyl)-, (T-4)- (9CI) (CA INDEX NAME).

PAGE 1-A

$$\begin{array}{c|c} F & \text{OH-Me} \\ \hline F & C & B & R2 \\ \hline F & F & C & F \\ \hline \end{array}$$

PAGE 2-A

$$F \longrightarrow F \longrightarrow F$$

$$F \longrightarrow F$$

$$R2 \longrightarrow C \longrightarrow F$$

RN 400644-13-5 CAPLUS

CN Boron, [2-[bis(pentafluorophenyl)boryl]-3,4,5,6tetrafluorophenyl](methanol)bis(pentafluorophenyl)-, (T-4)-, compd. with methanol (1:1) (9CI) (CA INDEX NAME)

CM 1

CRN 400644-12-4 CMF C31 H4 B2 F24 O CCI CCS

PAGE 1-A

$$F$$
 F
 F
 F

PAGE 2-A

CM 2

CRN 67-56-1 CMF C H4 O

H₃C-OH

Reaction of $[Ph3C] + [C6F4-1,2-\{B(C6F5)2\}2(.mu.-OCH3)]-$, 1, with Bu3SnH AΒ gives the solvated stannylium ion [Bu3Sn(arene)]+[C6F4-1,2- $\{B(C6F5)2\}2(.mu.-OCH3)\}$ -, 2, as a light brown oil (.delta. 119Sn = 434 ppm). This material is thermodynamically stable toward transfer of the chelated OMe- anion to "Bu3Sn+" as evidenced by the reaction of free diborane C6F4-1,2-[B(C6F5)2]2 with 2 equiv. of Bu3SnOMe. This reaction produces the stannyloxonium complex [(Bu3Sn)2OCH3]+[C6F4-1,2- $\{B(C6F5)2\}2(.mu.-OCH3)\}-$, 3 (.delta. 119Sn = 277 ppm), which is stable at -60.degree.. Upon warming, the cation in 3 undergoes decompn. to unidentified products, while the anion remains intact. Ion pair 2 reacts rapidly with ethereal HCl in CH2Cl2 to generate Bu3SnCl and the oxonium acid $[(Et20)2H]+[C6F4-1,2-\{B(C6F5)2\}2(.mu.-OCH3)]-$, 4, isolated in 76% yield as a white, cryst. solid. Acid 4 is thermally stable in soln. and was characterized crystallog. The C2B2OMe core of the anion in 4 deviates from planarity due to intermol. interactions in the crystal, in contrast to the structures found in other ion pairs with this anion. Reaction of 2 with anhyd. HCl gives the unsym. MeOH adduct of C6F4-1,2-[B(C6F5)2]2, 5, which was sep. synthesized by direct reaction of methanol with the free diborane. The anion $[C6F4-1,2-\{B(C6F5)2\}2(.mu.-OCH3)]$ is not stable in the presence of the triethylsilylium ion, generated from 1 and Et3SiH.

RE.CNT 36 THERE ARE 36 CITED REFERENCES AVAILABLE FOR THIS RECORD ALL CITATIONS AVAILABLE IN THE RE FORMAT

L4 ANSWER 35 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

AN 2001:872310 CAPLUS

DN 136:200214

TI Synthesis and structural studies on fluorophenylboron azides

AU Fraenk, Wolfgang; Klapotke, Thomas M.; Krumm, Burkhard; Mayer, Peter; Noth, Heinrich; Piotrowski, Holger; Suter, Max

CS Department of Chemistry, University of Munich, Munich, D-81377, Germany

SO Journal of Fluorine Chemistry (2001), 112(1), 73-81

CODEN: JFLCAR; ISSN: 0022-1139

PB Elsevier Science S.A.

DT Journal

LA English

IT 401844-91-5P

RN 401844-91-5 CAPLUS

CN Borinic acid, bis[2,4,6-tris(trifluoromethyl)phenyl]-, compd. with azidobis[2,4,6-tris(trifluoromethyl)phenyl]borane (1:1) (9CI) (CA INDEX NAME)

CM 1

CRN 401844-84-6 CMF C18 H4 B F18 N3

CM 2

CRN 192823-38-4 CMF C18 H5 B F18 O

The fluorophenyl substituted boron chlorides (RF) 2BCl and dichlorides RFBCl2 (RF = 2,6-F2C6H3, 2-FC6H4) were prepd. using stannylated aryl transfer reagents (RF) 2SnMe2. The boron azides (RF) 2BN3, [2,4,6-(CF3)3C6H2] 2BN3, and diazides RFB(N3)2 were synthesized by the reaction of the corresponding boron chlorides (RF) 2BCl, [(CF3)3C6H2] 2BCl, and RFBCl2 with Me3SiN3. The influence of the electron withdrawing substituents on the mol. structure of these azides is discussed. The reactions were also performed in the presence of pyridine yielding the adducts (RF) 2BN3.cntdot.py and RFB(N3) 2.cntdot.py. All compds. were characterized by multinuclear NMR, vibrational (IR, Raman) spectroscopy; and the mol. structures of several were established by single crystal x-ray crystallog.

RE.CNT 33 THERE ARE 33 CITED REFERENCES AVAILABLE FOR THIS RECORD ALL CITATIONS AVAILABLE IN THE RE FORMAT

L4 ANSWER 36 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN AN 2001:744009 CAPLUS

2622-89-1 CAPLUS

```
DN
     137:10926
TI
     Crosslink formation in porcine valves stabilized by dye-mediated
     photooxidation
     Adams, A. K.; Talman, E. A.; Campbell, L.; McIlroy, B. K.; Moore, M. A.
ΑU
     Sulzer Carbomedics, Austin, TX, 78752-1793, USA
CS
     Journal of Biomedical Materials Research (2001), 57(4), 582-587
SO
     CODEN: JBMRBG; ISSN: 0021-9304
     John Wiley & Sons, Inc.
PB
DT
     Journal
LΑ
     English
IT
     2622-89-1, Di-phenyl borinic acid
     RL: BSU (Biological study, unclassified); MOA (Modifier or additive use);
     THU (Therapeutic use); BIOL (Biological study); USES (Uses)
        (crosslink formation in porcine valves stabilized by dye-mediated
        photooxidn.)
```

Borinic acid, diphenyl- (6CI, 7CI, 8CI, 9CI) (CA INDEX NAME)

RN

CN

AΒ Bovine pericardial and porcine valve materials stabilized by dye-mediated photooxidn. have shown potential for bioprosthetic valve use. Previously, in vitro and in vivo stability of these materials was demonstrated through enzymic, chem., extn., rat s.c., and functional challenges. Here, we examine the stability of photooxidized porcine aortic valves through amino acid, crosslink, and hydrothermal isometric tension anal. Photooxidn. reduced intact histidine residues from 17.0 to 0 residues per 1000, indicating the photooxidative alteration of this amino acid. Di-Ph borinic acid-derivatized hydrolyzates of proteins were sepd. by high-performance liq. chromatog., which identified several amino acid crosslinks that appeared with photooxidn. that were absent in untreated controls. Thermal relaxation anal. indicated a significantly higher (p < 0.0002) thermal stability for photooxidized porcine cusps than that of untreated controls, with mean relaxation times for untreated cusps of 14,000.+-.4650 vs: 22,900.+-.2480 s for photooxidized cusps. In summary, porcine aortic valve tissue treated by dye-mediated photooxidn. contains new chem. species and exhibits properties consistent with intermol. crosslink formation, which explain the increased biostability of this material and its potential for use in bioprosthetic devices.

RE.CNT 27 THERE ARE 27 CITED REFERENCES AVAILABLE FOR THIS RECORD ALL CITATIONS AVAILABLE IN THE RE FORMAT

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L4 ANSWER 37 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN AN 2001:729439 CAPLUS
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DN 136:144922

- TI Toxicity and hypotensive effect of L-arginine oxoborolidinone and its modulation by methylene blue as compared to L-arginine, nitrite and nitrate
- AU Avila, Martha Elena Bravo; Alvarez, Juan Manuel Araujo; Quezada, Arturo Bustamante; Ferrara, Jose Guadalupe Trujillo
- CS Depto. de Bioquimica, Lab. de Bioquimica Medica I, Escuela Superior de Medicina, Mexico, DF, 11340, Mex.
- SO Archivos de Cardiologia de Mexico (2001), 71(3), 193-198 CODEN: ACMRCR; ISSN: 1405-9940

```
PB Instituto Nacional de Cardiologia "Ignacio Chavez"
DT Journal
LA Spanish
```

IT 2622-89-1, Diphenylborinic acid
RL: RCT (Reactant); RACT (Reactant or reagent)
(L-arginine oxoborolidinone hypotensive and toxic effects and their modulation by methylene blue as compared to L-arginine, Na nitrite and Na nitrate in Wistar rats)

RN 2622-89-1 CAPLUS

CN Borinic acid, diphenyl- (6CI, 7CI, 8CI, 9CI) (CA INDEX NAME)

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Ph
|
Ph— B— OH
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AB NO is synthesized by constitutive nitric oxide synthase from the guanidine group of L-arginine. The hypotensive effects of L-arginine oxoborolidinone were compared with the effects of L-arginine HCl, Na nitrite, and Na nitrate in male and female Wistar rats (250-300 g). The modulation of the L-arginine oxoborolidinone hypotensive effects by methylene blue, a synthetic phenothiazine inhibitor of guanylate cyclase, was examd. L-arginine, L-arginine oxoborolidinone, nitrite, and nitrate showed dose-dependent hypotensive effects after injection into the femoral vein of the rats. The hypotensive effects were shifted to the right after treatment with methylene blue. The L-arginine oxoborolidinone toxicity LD50 value was 169.0 mg/kg body wt. Methylene blue also attenuated the toxic effects of all the compds. tested.

RE.CNT 16 THERE ARE 16 CITED REFERENCES AVAILABLE FOR THIS RECORD ALL CITATIONS AVAILABLE IN THE RE FORMAT

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L4 ANSWER 38 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN
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AN 2001:718674 CAPLUS

DN 136:20104

TI 1H and 19F NMR Investigation of the Reaction of B(C6F5)3 with Water in Toluene Solution

AU Beringhelli, Tiziana; Maggioni, Daniela; D'Alfonso, Giuseppe

CS Dipartimento di Chimica Inorganica Metallorganica e Analitica, Milan, 20133, Italy

SO Organometallics (2001), 20(23), 4927-4938 CODEN: ORGND7; ISSN: 0276-7333

PB American Chemical Society

DT Journal

LA English

IT 155962-44-0 155962-45-1, Aquatris(pentafluorophenyl)boro
n 312640-05-4

RL: CPS (Chemical process); FMU (Formation, unclassified); PEP (Physical, engineering or chemical process); PRP (Properties); RCT (Reactant); FORM (Formation, nonpreparative); PROC (Process); RACT (Reactant or reagent) (proton and fluorine NMR investigation of reaction of tris(pentafluorophenyl)borane with water in toluene soln.)

RN 155962-44-0 CAPLUS

CN Boron, aquatris(pentafluorophenyl)-, dihydrate, (T-4)- (9CI) (CA INDEX NAME)

Patel 9/24/2003>

●2 H₂O

RN 155962-45-1 CAPLUS
CN Boron, aquatris(pentafluorophenyl)-, (T-4)- (9CI) (CA INDEX NAME)

$$F \xrightarrow{C^{-}} B \xrightarrow{3+} C \xrightarrow{F} F$$

$$F \xrightarrow{F} F$$

$$F \xrightarrow{F} F$$

RN 312640-05-4 CAPLUS .
CN Boron, aquatris(pentafluorophenyl)-, monohydrate, (T-4)- (9CI) (CA INDEX

H20

AB Titrns. of B(C6F5)3 (1) with water, in toluene-d8 soln., monitored by 19F and 1H NMR at 196 K, showed first the formation of the adduct [(C6F5)3B(OH2)] (2) and then its stepwise transformation into the two aqua species [(C6F5)3B(OH2)].H2O (3) and [(C6F5)3B(OH2)].2H2O (4) contg., resp., one or two water mols. hydrogen-bonded to the protons of the B-bound water mol. The NMR data show that in each titrn. step only two species were present in significant concn.: 1 and 2 up to 1 equiv., 2 and 3 between 1 and 2 equiv., 3 and 4 between 2 and 3 equiv. Above 3 equiv. the solns. rapidly attained satn. and phase sepn. occurred (although there was evidence of interaction of 4 with more water mols.). Titrns. at room temp. indicated an analogous stepwise course. Variable-temp. expts. demonstrated water exchange between the different agua species and between the different water sites in the adducts 3 and 4 ("internal" or B-bound and "external" or H-bound). The rate of these processes increased with the amt. of water bonded to B(C6F5)3. The exchange of B-bound water among the different B(C6F5)3 mols. (resulting in the 1 .dblharw. 2 interconversion) caused the averaging of the 19F resonances of 1 and 2, above 273 K. Band shape anal. in the temp. range 235-312 K provided the kinetic consts., whose dependence on the concn. revealed a dissociative mechanism (.DELTA.H.thermod. 67(2) kJ mol-1, .DELTA.S.thermod. 58(7) J mol-1 K-1). For the adduct [(C6F5)3B(OH2)].H2O (3), four different dynamic processes have been recognized: (i) the exchange of H-bound water among different [(C6F5)3B(OH2)] adducts (the 2 .dblharw. 3 exchange) or (ii) among different [(C6F5)3B(OH2)].H2O adducts (the 3 .dblharw. 4 exchange), (iii) the exchange between H-bound and B-bound water, (iv) the hopping of H-bound water between the two protons of B-bound water. This process was so fast that an averaged signal for the protons of internal water was obsd. even at 187 K. The rate of the process (i) increased with the concn. of 2, so that sep. 19F and 1H signals for 2 and 3 were obsd. only in very dil. solns. at the lowest temps. Linear plots of the kinetic consts. (estd. from 1H NMR spectra in the near fast exchange region, temp. range 188-214 K) vs. the concn. of 2 allowed the estn. of the const. for the dissociative pathway (4 orders of magnitude faster than for the exchange of B-bound water) and for the bimol. pathway [.DELTA.H.thermod. 30(2) kJ mol-1, .DELTA.S.thermod. 3(10) J mol-1 K-1]. Process (ii) was too fast on the NMR time scale to allow any kinetic investigation. Process (iii) caused the parallel broadening of both the 1H signals of 3 at T > 225 K, with a rate quite close to that of the dissociative exchange of water among different B(C6F5)3 mols. The activation parameters (.DELTA.H.thermod. 55(2) kJ mol-1, .DELTA.S.thermod. 7(3) J mol-1 K-1, temp. range 233-273 K) allowed no discrimination between the exchange of an entire water mol. and the mere exchange of protons. Even small amts. of 4 accelerated process (iii), due to the occurrence of two much faster processes: the 3 .dblharw. 4 exchange and the exchange between the protons of internal and external water in 4. The study of any kind of water mobility concerning the trihydrate 4 was prevented by the occurrence of proton exchange processes (so fast as to broaden the signals of internal and external water even at 188 K), possibly favored by the acidic dissocn. of the protons of the B-bonded water mol. of 4.

RE.CNT 71 THERE ARE 71 CITED REFERENCES AVAILABLE FOR THIS RECORD ALL CITATIONS AVAILABLE IN THE RE FORMAT

- L4 ANSWER 39 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN
- AN 2001:663995 CAPLUS
- DN 135:365956
- TI Characterization and Bonding of the Cation [Ge $\{N(C6H3-2,6-i-Pr2)CMe\}2CH\}+:$ Comparison with the Isoelectronic Ga $\{N(C6H3-2,6-i-Pr2)CMe\}2CH\}$
- AU Stender, Matthias; Phillips, Andrew D.; Power, Philip P.

Patel 9/24/2003>

Page 68

CS Department of Chemistry, University of California Davis, Davis, CA, 95616, USA

SO Inorganic Chemistry (2001), 40(21), 5314-5315 CODEN: INOCAJ; ISSN: 0020-1669

PB American Chemical Society

DT Journal

LA English

IT 373383-59-6P

RL: PRP (Properties); SPN (Synthetic preparation); PREP (Preparation) (prepn. and electronic and crystal structure and)

RN 373383-59-6 CAPLUS

CN Germanium(1+), [[N,N'-(1,3-dimethyl-1,3-propanediylidene)bis[2,6-bis(1-methylethyl)benzenaminato-.kappa.N]](1-)]-, .mu.-hydroxyhexakis(pentafluorophenýl)diborate(1-) (9CI) (CA INDEX NAME)

CM 1

CRN 373383-58-5 CMF C29 H41 Ge N2 CCI CCS

CM 2

CRN 219697-03-7 CMF C36 H B2 F30 O CCI CCS

PAGE 1-A

PAGE 2-A

$$F = \begin{bmatrix} F & F \\ F & F \end{bmatrix}$$

PAGE 3-A

AB Reaction of Ge(Cl)Dipp2nacnac (Dipp2nacnac = $\{N(C6H3-2,6-i-Pr2)C(Me)\}2CH\}$ with B(C6F5)3 in the presence of H2O affords the salt $[Ge(Dipp2nacnac)][HO\{B(C6F5)3\}2]$ (I) whose cation [Ge(Dipp2nacnac)]+ is isoelectronic to its neutral gallium analog Ga(Dipp2nacnac). I is monoclinic, space group P21/c, Z=4, R1=0.0561.

RE.CNT 23 THERE ARE 23 CITED REFERENCES AVAILABLE FOR THIS RECORD ALL CITATIONS AVAILABLE IN THE RE FORMAT

L4 ANSWER 40 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

AN 2001:657548 CAPLUS

DN 135:211145

TI Procedure for the production of mono or di-organo-boranes

IN Kratzer, Roland

PA Basell Polyolefine G.m.b.H., Germany

SO Ger. Offen., 6 pp. CODEN: GWXXBX

DT Patent

LA German

FAN.CNT 1

PATENT NO. KIND DATE APPLICATION NO. DATE

PI DE 10059717 A1 20010906 DE 2000-10059717 20001130

DE 2000-10009650A120000301

OS CASREACT 135:211145; MARPAT 135:211145

IT 2118-02-7P, Bis(pentafluorophenyl)borinic acid 2622-89-1P
73774-44-4P 73774-45-5P 89566-59-6P
176913-70-5P 279216-31-8P 357437-65-1P

357437-66-2P

RN 2118-02-7 CAPLUS

CN Borinic acid, bis(pentafluorophenyl) - (7CI, 8CI, 9CI) (CA INDEX NAME)

RN 2622-89-1 CAPLUS

CN Borinic acid, diphenyl- (6CI, 7CI, 8CI, 9CI) (CA INDEX NAME)

RN 73774-44-4 CAPLUS

CN Borinic acid, bis(2-methylphenyl) - (9CI) (CA INDEX NAME)

RN 73774-45-5 CAPLUS

CN Borinic acid, bis(4-methoxyphenyl)- (9CI) (CA INDEX NAME)

RN 89566-59-6 CAPLUS

CN Borinic acid, bis(4-chlorophenyl) - (9CI) (CA INDEX NAME)

RN 176913-70-5 CAPLUS

CN Borinic acid, bis(4-fluorophenyl) - (9CI) (CA INDEX NAME)

RN 279216-31-8 CAPLUS

CN Borinic acid, bis([1,1'-biphenyl]-4-yl)- (9CI) (CA INDEX NAME)

RN 357437-65-1 CAPLUS

CN Borinic acid, bis(2,2',3,3',4',5,5',6,6'-nonafluoro[1,1'-biphenyl]-4-yl)- (9CI) (CA INDEX NAME)

RN 357437-66-2 CAPLUS

CN Borinic acid, bis[4-(trifluoromethyl)phenyl]- (9CI) (CA INDEX NAME)

AB The present invention concerns a new, tech. feasible procedure for the prodn. of mono or di-organo-boranes, (C6R15)1+1M(XR9p)2-1 (R1 = same or

different, H, halo, O-, S-, or N- contg. C1-20-alkyl, C6-14-aryl, C2-10-alkenyl, C7-20-arylalkyl, C7-20-alkylaryl, C1-10-haloalkyl, C6-10-haloaryl, C2-10-alkynyl, C3-20-alkylsilyl, etc., M = Group III main group element, X = same or different, Group V or VI main group element, R9 = H, O-, S-, N- contg. C1-20-alkyl, C6-14-aryl, P = P-1-2, P = P-1). Thus, reaction of P-13 with P-propanol followed by treatment with (pentafluorophenyl) magnesium bromide gave title compd., P-15 bis (pentafluorophenyl) boronate.

L4 ANSWER 41 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

AN 2001:657472 CAPLUS

DN 135:211143

TI Procedure for the production of mono or di-organo-boranes

IN Schottek, Joerg; Fritze, Cornelia

PA Targor G.m.b.H., Germany

SO Ger. Offen., 6 pp. CODEN: GWXXBX

DT Patent

LA German

FAN.CNT 1

ΡI

PATENT NO. KIND DATE APPLICATION NO. DATE

DE 10009714 A1 20010906 DE 2000-10009714 20000301

DE 2000-10009714 20000301

OS CASREACT 135:211143; MARPAT 135:211143

IT 2118-02-7P 2622-89-1P 73774-44-4P 73774-45-5P 89566-59-6P 279216-31-8P 357437-65-1P 357437-66-2P

RN 2118-02-7 CAPLUS

CN Borinic acid, bis(pentafluorophenyl) - (7CI, 8CI, 9CI) (CA INDEX NAME)

RN 2622-89-1 CAPLUS

CN Borinic acid, diphenyl- (6CI, 7CI, 8CI, 9CI) (CA INDEX NAME)

RN 73774-44-4 CAPLUS

CN Borinic acid, bis(2-methylphenyl) - (9CI) (CA INDEX NAME)

RN 73774-45-5 CAPLUS

CNBorinic acid, bis(4-methoxyphenyl) - (9CI) (CA INDEX NAME)

RN 89566-59-6 CAPLUS

CN Borinic acid, bis(4-chlorophenyl) - (9CI) (CA INDEX NAME)

RN279216-31-8 CAPLUS

CN Borinic acid, bis([1,1'-biphenyl]-4-yl)- (9CI) (CA INDEX NAME)

RN

357437-65-1 CAPLUS
Borinic acid, bis(2,2',3,3',4',5,5',6,6'-nonafluoro[1,1'-biphenyl]-4-yl)-CN(9CI) (CA INDEX NAME)

RN 357437-66-2 CAPLUS

CN Borinic acid, bis[4-(trifluoromethyl)phenyl]- (9CI) (CA INDEX NAME)

The present invention concerns a new, tech. well feasible procedure for the prodn. of mono or di-organo-boranes, (C6R15)1+1M(XR9p)2-1 (R1 = same or different H, halo, O-, S-, or N- contg. C1-20-alkyl, C6-14-aryl, C2-10-alkenyl, C7-20-arylalkyl, C7-20-alkyaryl, C1-10-haloalkyl, C6-10-haloaryl, C2-10-alkynyl, C3-20-alkylsilyl, etc., M = main group element, X = Group V, VI main group element, R9 = H, O-, S-, N- contg. C1-20-alkyl, C6-14-aryl, p = 1-2). Thus, lithiation of bromopentafluorobenzene with BuLi in hexane followed by treatment with BC13 in hexane gave 96% bis(pentafluorophenyl)hydroxyborane.

L4 ANSWER 42 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

AN 2001:621596 CAPLUS

DN . 135:344095

TI Catalytic use of a boron source for boron enolate mediated stereoselective aldol reactions in water

AU Mori, Yuichiro; Manabe, Kei; Kobayashi, Shu

CS Graduate School of Pharmaceutical Sciences, The University of Tokyo, CREST Japan Science and Technology Corporation (JST), Tokyo, 113-0033, Japan

SO Angewandte Chemie, International Edition (2001), 40(15), 2815-2818 CODEN: ACIEF5; ISSN: 1433-7851

PB Wiley-VCH Verlag GmbH

DT Journal

LA English

IT 2622-89-1, Diphenylborinic acid

RL: CAT (Catalyst use); PEP (Physical, engineering or chemical process); RCT (Reactant); PROC (Process); RACT (Reactant or reagent); USES (Uses) (stereoselective Mukaiyama aldol reaction of aldehydes with silyl enol ethers in water under conditions of Lewis acid surfactant combined catalysis with Bronsted acids to facilitate Si-B exchange)

RN 2622-89-1 CAPLUS

CN Borinic acid, diphenyl- (6CI, 7CI, 8CI, 9CI) (CA INDEX NAME)

The stereoselective Mukaiyama aldol reaction of cis-propiophenone trimethysilyl enolate with PhCHO was performed in water under conditions of Lewis acid surfactant combined catalysis (using diphenylborinic acid 1 and SDS) in the presence of benzoic acid: the best yield (93%) and syn selectivity (syn/anti = 94/6) were obtained when 1 (0.1 equiv), SDS (0.1 equiv), and benzoic acid (0.01 equiv) were used at 0.degree. C. The intermediacy of a boron enolate was discussed, as well as the role of Bronsted acid (benzoic acid) in facilitating Si-B exchange.

RE CNT 24 THERE ARE 24 CITED REFERENCES AVAILABLE FOR THIS RECORD ALL CITATIONS AVAILABLE IN THE RE FORMAT

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L4
    ANSWER 43 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN
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2001:557142 CAPLUS AN

135:288814 DN

Unusual products in reactions using ethyldimesitylborane, mesityllithium, ΤI and carbonyl compounds

Kawashima, Takayuki; Yamashita, Naoko; Kannabe, Tohru; Okazaki, Renji ΑU

CS Department of Chemistry, Graduate School of Science, The University of Tokyo, Tokyo, 113-0033, Japan

SO Heteroatom Chemistry (2001), 12(5), 354-357 CODEN: HETCE8; ISSN: 1042-7163

PB . John Wiley & Sons, Inc.

DT Journal

English LΑ

CASREACT 135:288814 OS

ΙT 20631-84-9P

RL: SPN (Synthetic preparation); PREP (Preparation) (prepn. of)

RN 20631-84-9 CAPLUS

CN Borinic acid, bis(2,4,6-trimethylphenyl) - (9CI) (CA INDEX NAME)

AΒ Unusual carbonyl adducts, (E)-Mes2BCH:CHCHCH3CRR1OH (Mes = 2,4,6-Me3C6H2, R, R1 = Ph, CH2Ph), were obtained by sequential treatment of ethyldimesitylborane with mesityllithium (<1.0 equiv) and carbonyl compds., e.g., RR1CO, instead of the normal adducts, Mes2BCHCH3CRR1OH. A mechanistic study was carried out.

RE.CNT 21 THERE ARE 21 CITED REFERENCES AVAILABLE FOR THIS RECORD ALL CITATIONS AVAILABLE IN THE RE FORMAT

L4ANSWER 44 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

ΑN 2001:479135 CAPLUS

DN 135:77275

TIMetallocene catalyst system and its use for polymerization of olefins

INKratzer, Roland; Fritze, Cornelia; Schottek, Joerg

PA Targor G.m.b.H., Germany

SO Ger. Offen., 18 pp. CODEN: GWXXBX

DT Patent

LΑ German

FAN.CNT 1																		
	PATENT NO.			KI	ND	DATE			Α	PPLI	CATI	ON NO	Ο.	DATE				
										-								
ΡI	DE	1996	52814		Α	1	2001	0628		D:	E 19	99-1	9962	814	1999	1223		
	WO	200	L0476	35	A.	2	2001	0705		W	O 20	00-E	P126	41	2000	1213		
	WO 2001047635		A.	3	2002	1024												
		W:	ΑE,	AG,	AL,	AM,	AT,	AU,	ΑZ,	BA,	BB,	BG,	BR,	BY,	BZ,	CA,	CH,	CN,
			CR,	CU,	CZ,	DE,	DK,	DM,	DZ,	EE,	ES,	FI,	GB,	GD,	GE,	GH,	GM,	HR,
			ΗU,	ID,	IL,	IN,	IS,	JP,	KE,	KG,	ΚP,	KR,	ΚZ,	LC,	LK,	LR,	LS,	LT,
			LU,	LV,	MA,	MD,	MG,	MK,	MN,	MW,	MX,	MZ,	NO.	NZ,	PL,	PT.	RO.	RU.

Patel 9/24/2003>

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SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, TZ, UA, UG, US, UZ, VN,
             YU, ZA, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM
         RW: GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZW, AT, BE, CH, CY,
             DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, TR, BF,
             BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG
                                           DE 1999-19962814A 19991223
    BR 2000016725
                            20020903
                                           BR 2000-16725
                                                            20001213
                                           DE 1999-19962814A 19991223
                                           WO 2000-EP12641W 20001213
    EP 1280600
                            20030205
                                           EP 2000-983307
                       A2
                                                           20001213
           AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT,
             IE, SI, LT, LV, FI, RO, MK, CY, AL, TR
                                           DE 1999-19962814A 19991223
                                           WO 2000-EP12641W 20001213
                            20030109
    US 2003008984
                       A1
                                           US 2002-168646
                                                            20020624
                                           DE 1999-19962814A 19991223
                                           WO 2000-EP12641W 20001213
OS
    MARPAT 135:77275
IT
    2118-02-7, Bis (pentafluorophenyl) borinic acid
    RL: RCT (Reactant); RACT (Reactant or reagent)
        (supported metallocene catalyst system for polymn. of olefins)
    2118-02-7 CAPLUS
RN
CN
    Borinic acid, bis(pentafluorophenyl) - (7CI, 8CI, 9CI) (CA INDEX NAME)
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The catalyst system, which avoids the use of a large excess of aluminoxane, contains .gtoreq.1 metallocene, .gtoreq.1 Lewis base, .gtoreq.1 carrier and .gtoreq.1 addnl. organometallic compd. Thus, reaction of Me3Al with C6F5B(OH)2 in toluene gave C6F5B(OAlMe2)2 (I). A suspension of SiO2 in toluene was treated with PhCH2NMe2 at room temp. and then with I at .apprx.0.degree. to give a support, which was mixed with [(dimethylsilylene)bis(2-methyl-4-phenylindenyl)]dimethylzirconium and then Me3Al to give a catalyst powder. Polymn. of liq. propylene (pretreated with iso-Bu3Al) with the catalyst powder in heptane at 60.degree. gave 28 kg polypropylene/g metallocene per h.

- L4 ANSWER 45 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN
- AN 2001:447350 CAPLUS
- DN 135:223264
- TI Carbonic anhydrase inhibitors, interaction of boron derivatives with isozymes I and II: a new binding site for hydrophobic inhibitors at the entrance of the active site as shown by docking studies
- AU Chazalette, Celine; Riviere-Baudet, Monique; Scozzafava, Andrea; Abbate, Francesco; Maarouf, Zahra Ben; Supuran, Claudiu T.
- CS Universite Paul Sabatier, Laboratoire d'Heterochimie Fondamentale et Appliquee, UMR 5069 du CNRS, Toulouse, 31062, Fr.
- SO Journal of Enzyme Inhibition (2001), 16(2), 125-133 CODEN: ENINEG; ISSN: 8755-5093

PB Harwood Academic Publishers

DT Journal

LA English

OS CASREACT 135:223264

IT 20631-84-9P

RL: BAC (Biological activity or effector, except adverse); BSU (Biological study, unclassified); PRP (Properties); SPN (Synthetic preparation); BIOL (Biological study); PREP (Preparation)

(prepn. and interaction of sulfonamide and non-sulfonamide boron derivs. with carbonic anhydrase isoenzymes I and II)

RN 20631-84-9 CAPLUS

CN Borinic acid, bis(2,4,6-trimethylphenyl) - (9CI) (CA INDEX NAME)

AΒ The interaction of human carbonic anhydrase (hCA) isoenzymes I and II with boron derivs. was investigated by kinetic and spectroscopic studies. These derivs., tested as new inhibitors of carbonic anhydrase, are sulfonamide and non-sulfonamide boron derivs. and some of them proved to be moderately efficient inhibitors of hCA I and hCA II, their activities being comparable to those of the unsubstituted sulfonamides, the classical inhibitors of these zinc enzymes. Ph2BOH, one of the compds. with the highest affinity for hCA II in the present study, has been docked within the active site. After minimization it was found situated at 7.9 .ANG. from zinc, within the hydrophobic half of the active site, in Van der Waals contacts with the amino acid residues: Val 121; Phe 130, Val 135, Leu 141, Val 143, Val 207 and Pro 201. This is the first time that a CA inhibitor has been found to bind at the edge of the active site cavity, similarly to the CA activator histamine, which binds on the hydrophilic half. This finding may be of importance also for the design of novel types of inhibitors with increased affinity for the different CA isoenzymes.

RE.CNT 45 THERE ARE 45 CITED REFERENCES AVAILABLE FOR THIS RECORD ALL CITATIONS AVAILABLE IN THE RE FORMAT

L4 ANSWER 46 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

AN 2001:387341 CAPLUS

DN 135:137536

TI Suzuki Reaction of a Diarylborinic Acid: One-Pot Preparation and Cross-Coupling of Bis(3,5-dimethylphenyl)borinic Acid

AU Winkle, Derick D.; Schaab, Kevin M.

CS Holland Laboratories, Pfizer Global Research and Development, Holland, MI, 49424, USA

SO Organic Process Research & Development (2001), 5(4), 450-451 CODEN: OPRDFK; ISSN: 1083-6160

PB - American Chemical Society

DT Journal

LA English

OS CASREACT 135:137536

IT 352212-19-2P

RL: BYP (Byproduct); RCT (Reactant); SPN (Synthetic preparation); PREP

Patel 9/24/2003>

(Preparation); RACT (Reactant or reagent)

(one-pot prepn. and cross-coupling with cyclic vinyl triflate deriv.)

RN 352212-19-2 CAPLUS

CN Borinic acid, bis(3,5-dimethylphenyl) - (9CI) (CA INDEX NAME)

GΙ

AB 3,5-Dimethylphenylmagnesium bromide reacted with triisopropyl borate to give 3,5-dimethylphenylboronic acid and bis(3,5-dimethylphenyl)borinic acid. Conditions were found which allowed the clean prepn. of bis(3,5-dimethylphenyl)borinic acid, which was coupled with a vinyl triflate (I; R = OTf) using Suzuki cross-coupling conditions to give the arylated product (I; R = 3,5-dimethylphenyl) in 91% yield. Both aryl groups were efficiently transferred from the B atom in the Suzuki step.

RE.CNT 14 THERE ARE 14 CITED REFERENCES AVAILABLE FOR THIS RECORD ALL CITATIONS AVAILABLE IN THE RE FORMAT

L4 ANSWER 47 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

AN 2001:345136 CAPLUS

DN 135:12345

TI Dimethyl (2-methylphenyl) ammonium hydroxotris (pentafluorophenyl) borate

AU Stibrany, Robert T.; Brant, Patrick

CS Corporate Strategic Research, ExxonMobil Research and Engineering Company, Annandale, NJ, 08801, USA

SO Acta Crystallographica, Section C: Crystal Structure Communications (2001), C57(5), 644-645 CODEN: ACSCEE; ISSN: 0108-2701

PB Munksgaard International Publishers Ltd.

DT Journal

LA English

IT 341990-27-0

RL: PRP (Properties)
(crystal structure of)

RN 341990-27-0 CAPLUS

CN Borate(1-), hydroxytris(pentafluorophenyl)-, (T-4)-, hydrogen, compd. with N,N,2-trimethylbenzenamine (1:1) (9CI) (CA INDEX NAME)

CM 1

CRN 147892-17-9 CMF C18 H B F15 O . H CCI CCS

● н •

CM 2

CRN 609-72-3 CMF C9 H13 N

AB In the title compd., [Me2(C7H7)NH][(C6F5)3B(OH)] or C9H14N+.cntdot.C18HBF15O-, the distorted tetrahedral borate anions are strongly H bonded to the substituted ammonium cations. The N...O sepn. in the N-H...O H bond is 2.728(3) .ANG.. Crystallog. data are given.

RE.CNT 18 THERE ARE 18 CITED REFERENCES AVAILABLE FOR THIS RECORD

L4 ANSWER 48 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

ALL CITATIONS AVAILABLE IN THE RE FORMAT

AN 2001:320008 CAPLUS

DN 134:327285

TI Catalyst for polymerizing and/or crosslinking polyorganosiloxanes with crosslinkable functional groups, corresponding compositions and their uses

IN Frances, Jean-Marc; Deforth, Thomas

PA Rhodia Chimie, Fr.

SO PCT Int. Appl., 50 pp.

CODEN: PIXXD2

DT Patent

LA French

FAN.CNT 1

Page 80

	PATENT NO.	KIND DATE	APPLICATION NO.	DATE						
PI	W: AE, AG, CR, CU, HU, ID, LU, LV, SD, SE, YU, ZA, RW: GH, GM,	AL, AM, AT, AU, CZ, DE, DK, DM, IL, IN, IS, JP, MA, MD, MG, MK, SG, SI, SK, SL, ZW, AM, AZ, BY, KE, LS, MW, MZ,	WO 2000-FR2789 AZ, BA, BB, BG, BR, BY, DZ, EE, ES, FI, GB, GD, KE, KG, KP, KR, KZ, LC, MN, MW, MX, MZ, NO, NZ, TJ, TM, TR, TT, TZ, UA, KG, KZ, MD, RU, TJ, TM SD, SL, SZ, TZ, UG, ZW, GR, IE, IT, LU, MC, NL,	BZ, CA, CH, CN, GE, GH, GM, HR, LK, LR, LS, LT, PL, PT, RO, RU, UG, US, UZ, VN,						
	CF, CG,	CI, CM, GA, GN,	GW, ML, MR, NE, SN, TD, FR 1999-13620 A							
	FR 2800380	A1 20010504	FR 1999-13620							
	FR 2800380									
	EP 1226210	A1 20020731	EP 2000-967974	20001006						
	R: AT, BE,		FR, GB, GR, IT, LI, LU, MK, CY, AL	NL, SE, MC, PT,						
	•		FR 1999-13620 A 19991029 WO 2000-FR2789 W 20001006							
	JP 2003515617	T2 20030507		20001006 19991029						
os	MARPAT 134:3272	85	WO 2000-11(270) W	20001008						
IT										
RN	2118-02-7 CAPL		<u>.</u>							
CN			nyl)- (7CI, 8CI, 9CI)	(CA INDEX NAME)						

The invention relates to a heat-activated catalyst with activation temp. AB <150.degree. for polymg. and/or crosslinking polyorganosiloxane-type monomers, oligomers and/or polymers with organofunctional groups, comprising a boron deriv. of formula (I): (A)xB(R')y, wherein the symbols R' are the same or different and represent an alkyl or alkenyl radical in C1-C12, an alkoxy radical in C1-C12, a Ph radical substituted by at least one electron attractor element, an aryl radical contg. at least two arom. rings such as biphenyl, naphthyl, optionally substituted by at least one electron attracting group, esp. a halogen atom (particularly fluorine), or an electron attracting group, esp. a CF3, NO2, CN group; and a radical -C2H4-Si(Q)3 with the symbols Q being the same or different and representing an alkyl or alkoxy group in C1 to C10 or a siloxane oligomer with less than 10 silicon atoms. The invention also relates to a corresponding crosslinkable compn. and to the uses thereof. RE.CNT 2 THERE ARE 2 CITED REFERENCES AVAILABLE FOR THIS RECORD

ALL CITATIONS AVAILABLE IN THE RE FORMAT

L4 ANSWER 49 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

AN 2001:271432 CAPLUS

DN 135:61370

TI Structural studies of organoboron compounds. LXXIII. Formation of heterocyclic arylboronates by thermally induced 1,4-aryl migration in diarylboron chelates of C-(1-hydroxyalkyl)nitrones. Crystal and molecular structures of some of the rearrangement products and of one of the educts

AU Kliegel, Wolfgang; Lubkowitz, Gottfried; Pokriefke, Jens O.; Rettig, Steven J.; Trotter, James

CS Institut fur Pharmazeutische Chemie der Technischen Universitat Braunschweig, Braunschweig, 38106, Germany

SO Canadian Journal of Chemistry (2001), 79(2), 226-237 CODEN: CJCHAG; ISSN: 0008-4042

PB National Research Council of Canada

DT Journal

LA English

OS CASREACT 135:61370

IT 13331-25-4, B-(1-Naphthyl)-B-phenylborinic acid 66117-64-4 73774-45-5

RL: RCT (Reactant); RACT (Reactant or reagent) (cyclization of, with hydroxybutanone and benzhydrylhydroxylamine)

RN 13331-25-4 CAPLUS

CN Borinic acid, 1-naphthalenylphenyl- (9CI) (CA INDEX NAME)

RN 66117-64-4 CAPLUS

CN Borinic acid, bis(4-methylphenyl) - (9CI) (CA INDEX NAME)

RN 73774-45-5 CAPLUS

CN Borinic acid, bis(4-methoxyphenyl)- (9CI) (CA INDEX NAME)

GI

Patel

9/24/2003>

AΒ Thermally induced rearrangement of diarylboron chelates of C-(1-hydroxyalkyl)nitrones I (R = me, CMe3, CH2Ph, CHPh2, Pr, CHMe2, cyclohexyl, 4-tolyl; R1 = H, Me; R2 = Ph, 4-tolyl, 4-MeOC6H4, 4-ClC6H4 la-n) gave 44-86% heterocyclic arylboronates II (same R-R2, 2a-n). The structures were detd. from spectroscopic data and from x-ray analyses. Thermoanal. and time-dependent NMR measurements give information on the nature of the enthalpy and kinetics of the isomerization reaction. Crystal data (at 293 K for 2j, 180 K for the others): 1m (R = CHPh2, R1 = Me, R2 = 4-ClC6H4), monoclinic, space group P21/n, a 14.059(3), b 12.5531(13), c 14.8531(6) .ANG., .beta. 95.8067(12).degree., Z = 4; 2j (R = CHPh2, R1 = Me, R2 = Ph), triclinic, space group P1, a 10.4729(11), b 13.5896(11), c 9.5803(7) .ANG., .alpha. 104.764(6), .beta. 103.279(7), .gamma. 107.278(7).degree., Z = 2; 2m (same R-R2 as 1m), monoclinic, space group P21/n, a 14.9442(13), b 11.990(2), c 16.0613(4) .ANG., .beta. 114.0153(7).degree., Z = 4; 9 (shown as III), monoclinic, P21/n, a11.123(2), b 18.433(3), c 13.4852(4) .ANG., .beta. 108.2075(7).degree., Z = 4. The structures were solved by direct methods and refined by full-matrix least-squares procedures to R(F, I .gtoreq. 3.sigma.(I)) = 0.036, 0.052, 0.043, and 0.040, resp., for AFC6 data for 2j and CCD data for 1m, 2m, and 9. All four mols. contain six-membered OBONCC rings, with an approx. planar ON:CC segment in the educt 1m and approx. planar OBOC segments in the rearrangement products. A probable transition state geometry is derived for the isomerization process.

RE.CNT 35 THERE ARE 35 CITED REFERENCES AVAILABLE FOR THIS RECORD ALL CITATIONS AVAILABLE IN THE RE FORMAT

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L4 ANSWER 50 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN
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AN 2001:228311 CAPLUS

DN 134:267814

TI Antifouling coating with trisubstituted borane-amine complex

IN Yamamori, Naoki; Nakamura, Isao; Kushi, Yoshinori; Yokoi, Junji; Arai, Tomokazu

PA Nippon Paint Co., Ltd., Japan

SO Eur. Pat. Appl., 20 pp. CODEN: EPXXDW

DT Patent

LA English

FAN.CNT 1

PATENT NO. KIND DATE APPLICATION NO. DATE

Page 83

EP 1999-307477 19990921 ΡI EP 1086996 20010328 A1 R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, SI, LT, LV, FI, RO

EP 1999-307477 19990921 12113-07-4DP, Triphenylborane-sodium hydroxide adduct, reaction IT products with amino-contg. polymer and benzaldehyde 331749-37-2DP , Diphenylmonooctylborane-sodium hydroxide adduct, reaction products with amino-contg. polymer and/or benzaldehyde RL: IMF (Industrial manufacture); POF (Polymer in formulation); PRP (Properties); TEM (Technical or engineered material use); PREP (Preparation); USES (Uses)

(antifouling coating with trisubstituted borane-amine complex)

12113-07-4 CAPLUS RN

Borate(1-), hydroxytriphenyl-, sodium, (T-4)- (9CI) (CA INDEX NAME) CN

Na+

RN 331749-37-2 CAPLUS Borate(1-), hydroxyoctyldiphenyl-, sodium, (T-4)- (9CI) (CA INDEX NAME) CN

Na+

GΙ

- AB The present invention is related to a resin for use in an antifouling coating which is a polymer obtained by polymg. a polymerizable unsatd. monomer(s) and having, at side chain terminals thereof, a trisubstituted borane-amine complex and an azomethine group, or a group represented by the following general formula I (wherein M=copper, zinc, nickel and cobalt, means a chem. bound state, W represents -N(R1)R2- (R1, R2=H, C1-4 alkyl group), -OCO-, -OSO2- or substituted pyridine, L=C1-4 alkylene group, m=0-4). Thus, an antifouling coating based on the reaction products of polyallylamine, triphenylborane-sodium hydroxide adduct and benzaldehyde, shows excellent effects over a long period of time.
- RE.CNT 4 THERE ARE 4 CITED REFERENCES AVAILABLE FOR THIS RECORD ALL CITATIONS AVAILABLE IN THE RE FORMAT
- L4 ANSWER 51 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN
- AN 2001:143647 CAPLUS
- DN 134:193208
- TI Preparation of nitrophenylphenol
- IN Kumamoto, Nobumitsu
- PA Hokko Chemical Industry Co., Ltd., Japan
- SO Jpn. Kokai Tokkyo Koho, 13 pp. CODEN: JKXXAF
- DT Patent
- LA Japanese

FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO. DATE				
PI	JP 2001055360	A2	20010227	JP 1999-294325 19991015				
				JP 1998-304423 A 19981026				
				TD 1000_150460 A 10000607				

- OS CASREACT 134:193208; MARPAT 134:193208
- IT 326926-55-0P

RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)

(prepn. of nitrophenylphenol by cross-coupling reaction of nitrohalobenzenes with phenylboronic acids and deprotection)

- RN 326926-55-0 CAPLUS
- CN Borinic acid, bis[4-(1,1-dimethylethoxy)phenyl] (9CI) (CA INDEX NAME)

- AB HOC6H4C6H4NO2 is prepd. by reaction of NO2C6H4X (X = halo) with (tert-BuOC6H4)3-nB(OH)n (n = 0-2) or tris(tert-butoxyphenyl)boroxin in the presence of bases and catalysts. P-nitroiodobenzene was reacted with tert-butoxyphenylboronic acid in the presence of Pd(OAc)2 and Ba(OH)2 in H2O-THF under reflux for 5 h and treated with H2SO4 in THF at 20-30.degree. for 2 h to give 76% 4-(p-nitrophenyl)phenol.
- L4 ANSWER 52 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN
- AN 2001:91533 CAPLUS
- DN 134:141720
- TI Boronic acid derivative inhibitors of .beta.-lactamases and antibacterial use

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IN
     Shoichet, Brian K.; Weston, Grady Scott
PΑ
    Northwestern University, USA
SO
    U.S., 30 pp., Cont.-in-part of U.S. 6,075,014.
     CODEN: USXXAM
DT
     Patent
     English
LΑ
FAN.CNT 2
     PATENT NO.
                     KIND DATE
                                          APPLICATION NO. DATE
PΙ
    US 6184363
                      B1
                            20010206
                                          US 1998-212851 19981216
                                          US 1997-49992P P 19970613
                                          US 1998-96893 A219980612
    US 6075014
                      A
                            20000613
                                          US 1998-96893 19980612
                                          US 1997-49992P P 19970613
    US 6417174
                      В1
                            20020709
                                          US 2000-587794
                                          US 1997-49992P P 19970613
                                          US 1998-96893 A319980612
    US 6448238
                      В1
                            20020910
                                          US 2000-620268
                                                           20000719
                                          US 1997-49992P P 19970613
                                          US 1998-96893 A219980612
                                          US 1998-212851 A319981216
PATENT FAMILY INFORMATION:
FAN 1999:7834
     PATENT NO.
                     KIND DATE
                                          APPLICATION NO.
                                                           DATE
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                                      WO 1998-US12096 19980612
    WO 9856392
PΙ
                     A1 19981217
        W: AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CU, CZ, DE,
             DK, EE, ES, FI, GB, GE, GH, GM, GW, HU, ID, IL, IS, JP, KE, KG,
             KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MD, MG, MK, MN, MW, MX,
             NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT,
             UA, UG, UZ, VN, YU, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM
        RW: GH, GM, KE, LS, MW, SD, SZ, UG, ZW, AT, BE, CH, CY, DE, DK, ES,
             FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, BF, BJ, CF, CG, CI,
             CM, GA, GN, ML, MR, NE, SN, TD, TG
                                          US 1997-49992P P 19970613
    AU 9881408
                      Α1
                            19981230
                                          AU 1998-81408
                                                          19980612
                                          US 1997-49992P P 19970613
                                          WO 1998-US12096W 19980612
     EP 1009415
                      A 1
                            20000621
                                          EP 1998-931233 19980612
        R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, NL, SE, PT, IE, FI
                                          US 1997-49992P P 19970613
                                          WO 1998-US12096W 19980612
    JP 2002504122
                      T2
                            20020205
                                          JP 1999-503192
                                                           19980612
                                          US 1997-49992P P 19970613
                                          WO 1998-US12096W 19980612
OS
    MARPAT 134:141720
IT
     2622-89-1 324024-77-3
     RL: BAC (Biological activity or effector, except adverse); BSU (Biological
     study, unclassified); THU (Therapeutic use); BIOL (Biological study); USES
        (boronic acid deriv. inhibitors of .beta.-lactamases and antibacterial
       use)
     2622-89-1 CAPLUS
RN
CN
    Borinic acid, diphenyl- (6CI, 7CI, 8CI, 9CI) (CA INDEX NAME)
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Patel 9/24/2003>

RN 324024-77-3 CAPLUS

CN Boronic acid, [4'-(hydroxyphenylboryl)[1,1'-biphenyl]-4-yl]- (9CI) (CA INDEX NAME)

AB The invention provides non-.beta.-lactam boronic acid deriv. inhibitors of .beta.-lactamases. The compds. may be used with .beta.-lactam antibiotics to treat .beta.-lactam-antibiotic-resistant bacterial infections. These compds. are also antibacterial by themselves. Finally, the invention provides a pharmaceutical compn. comprising these compds.

RE.CNT 36 THERE ARE 36 CITED REFERENCES AVAILABLE FOR THIS RECORD ALL CITATIONS AVAILABLE IN THE RE FORMAT

L4 ANSWER 53 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

AN 2001:90961 CAPLUS

DN 134:295853

TI Conformational analysis of boron-containing compounds using Gillespie-Kepert version of molecular mechanics

AU Otkidach, D. S.; Pletnev, I. V.

CS Chemistry Department, Lomonosov Moscow State University, Moscow, 119899, Russia

SO THEOCHEM (2001), 536(1), 65-72 CODEN: THEODJ; ISSN: 0166-1280

PB Elsevier Science B.V.

DT Journal

LA English

IT 20631-84-9 40905-43-9

RL: PEP (Physical, engineering or chemical process); PRP (Properties); PROC (Process)

(conformational anal. of boron-contg. compds. using Gillespie-Kepert version of mol. mechanics)

RN 20631-84-9 CAPLUS

CN Borinic acid, bis(2,4,6-trimethylphenyl)- (9CI) (CA INDEX NAME)

RN 40905-43-9 CAPLUS

CN Borate(1-), hydroxytriphenyl-, (T-4)- (9CI) (CA INDEX NAME)

AB Mol. mechanics force field for B-contg. compds. based on CHARMM parameters and Gillespie-Kepert (GK) model is developed. GK potential functions are applied to the coordination sphere of three-coordinated (uncharged) or four-coordinated (anionic) B atom. The force field provides an accurate description of exptl. x-ray stereochemistries in a wide range of organoboranes, organoborate complexes with polyhydroxy compds., mixed oligomers of boric acid and borates.

RE.CNT 16 THERE ARE 16 CITED REFERENCES AVAILABLE FOR THIS RECORD ALL CITATIONS AVAILABLE IN THE RE FORMAT

L4 ANSWER 54 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

AN 2001:85386 CAPLUS

DN 134:123802

TI [Hydroxytris(pentafluorophenyl)borato-0][N-isopropyl-2-(isopropylamino)troponiminato-N,N']methylindium

AU Guzei, Ilia A.; Delpech, Fabien; Jordan, Richard F.

CS Department of Chemistry, Iowa State University of Science and Technology, Ames, IA, 50011, USA

SO Acta Crystallographica, Section C: Crystal Structure Communications (2000), C56(8), E327-E328
CODEN: ACSCEE; ISSN: 0108-2701

PB Munksgaard International Publishers Ltd.

DT Journal

LA English

RN 320585-00-0 CAPLUS

CN Indium, [hydroxytris(pentafluorophenyl)borato(1-)-.kappa.O]methyl[N-(1-methylethyl)-7-[(1-methylethyl)imino-.kappa.N]-1,3,5-cycloheptatrien-1-aminato-.kappa.N]-, (T-4)- (9CI) (CA INDEX NAME)

AB Crystals of the title complex are monoclinic, space group P21/c, with a 19.6683(16), b 18.7739(16), c 19.8049(17) .ANG., .beta. 117.201(1).degree.; Z = 4 (2 mols./Z), dc = 1.761; R = 0.031, Rw(F2) = 0.075 for 13,220 reflections. The complex crystallizes as an ion pair linked by a .mu.-hydroxo bridge. The two independent mols. in the asym. unit exhibit essentially identical metric parameters, but different conformations.

RE.CNT 15 THERE ARE 15 CITED REFERENCES AVAILABLE FOR THIS RECORD ALL CITATIONS AVAILABLE IN THE RE FORMAT

L4 ANSWER 55 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

AN 2001:64049 CAPLUS

DN 134:132985

TI Triphenylboron-containing polymers and uses thereof in antifouling coatings

IN Yoshimaru, Masaaki; Kohara, Masanori; Shibuya, Yoshifumi

PA Yoshitomi Fine Chemicals, Ltd., Japan

SO PCT Int. Appl., 74 pp.

CODEN: PIXXD2

DT Patent

LA Japanese

FAN.CNT 1

1111	PATENT NO.	KIND DATE	APPLICATION NO.	DATE
PI	WO 2001005848 W: CN, JP,	A1 20010125 KR, NO, SG, US	WO 2000-JP4888	20000721
	RW: AT, BE, PT, SE	CH, CY, DE, DK, ES,	FI, FR, GB, GR, IE,	IT, LU, MC, NL,
			JP 1999-206799 A JP 2000-76939 A JP 2000-80153 A	20000317
	EP 1227111	A1 20020731	EP 2000-946441	20000721
	R: AT, BE, IE, FI,		GB, GR, IT, LI, LU,	NL, SE, MC, PT,
	·		JP 1999-206799 A JP 2000-76939 A JP 2000-80153 A	20000317

IT 12113-07-4P

WO 2000-JP4888 W 20000721

RL: IMF (Industrial manufacture); RCT (Reactant); PREP (Preparation); RACT (Reactant or reagent)

(triphenylboron-contg. vinyl polymers for antifouling coatings)

RN 12113-07-4 CAPLUS

CN Borate(1-), hydroxytriphenyl-, sodium, (T-4)- (9CI) (CA INDEX NAME)

Na +

AB Polymers contg. triphenylboron groups are used as active ingredients and binders. Thus, 2-ethylhexyl acrylate-methacrylic acid-Me methacrylate copolymer triphenylboron-ethylenediamine salt was prepd.

RE.CNT 3 THERE ARE 3 CITED REFERENCES AVAILABLE FOR THIS RECORD ALL CITATIONS AVAILABLE IN THE RE FORMAT

L4 ANSWER 56 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

AN 2001:53481 CAPLUS

DN 134:237539

TI Formation and Unexpected Catalytic Reactivity of Organoaluminum Boryloxides

AU Gibson, Vernon C.; Mastroianni, Sergio; White, Andrew J. P.; Williams, David J.

CS Department of Chemistry, Imperial College, London, SW7 2AY, UK

SO Inorganic Chemistry (2001), 40(5), 826-827 CODEN: INOCAJ; ISSN: 0020-1669

PB American Chemical Society

DT Journal

LA English

IT 20631-84-9, Dimesitylborinic acid 330157-33-0,

Bis(2,6-dimethylphenyl)borinic acid

RL: RCT (Reactant); RACT (Reactant or reagent)

(reactions with organoaluminum compds. and cyclocondensation catalyzed by aluminum boryloxo complexes)

RN 20631-84-9 CAPLUS

CN Borinic acid, bis(2,4,6-trimethylphenyl) - (9CI) (CA INDEX NAME)

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10085368.2
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Page 90

RN 330157-33-0 CAPLUS

CN Borinic acid, bis(2,6-dimethylphenyl) - (9CI) (CA INDEX NAME)

AB Reactions of Me2AlX (X = Me, Cl) with the borinic acids HOBR2 (R = mesityl or 2,6-dimethylphenyl) to give novel organoboryl oxide Al products are reported. An unexpected outcome of these studies was the finding that Al-OBR2 species are capable of catalyzing the formation of boroxine (RBO)3, a trimeric B relative of the hexanuclear aluminoxanes which may be viewed as being composed of two stacked (RAIO)3 rings. The crystal and mol. structures of [(.mu.-R2BO)2(AlMe2)2] (R = mesityl) and [(R2BO)3Al(NCMe)] were detd. by x-ray crystallog.

RE CNT 13 THERE ARE 13 CITED REFERENCES AVAILABLE FOR THIS RECORD ALL CITATIONS AVAILABLE IN THE RE FORMAT

L4 ANSWER 57 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

AN 2001:39789 CAPLUS

DN 134:295884

TI Triple-decker main group cations

AU Cowley, Alan H.; Macdonald, Charles L. B.; Silverman, Joel S.; Gorden, John D.; Voigt, Andreas

CS Department of Chemistry and Biochemistry, The University of Texas at Austin, Austin, TX, 78712, USA

SO Chemical Communications (Cambridge) (2001), (2), 175-176 CODEN: CHCOFS; ISSN: 1359-7345

PB Royal Society of Chemistry

DT Journal

LA English

IT 334023-90-4P

RL: PRP (Properties); SPN (Synthetic preparation); PREP (Preparation) (prepn. and crystal structure of)

RN 334023-90-4 CAPLUS

CN Indium(1+), bis[(1,2,3,4,5,6-.eta.)-methylbenzene][.mu.-[(1,2,3,4,5-.eta.:1,2,3,4,5-.eta.)-1,2,3,4,5-pentamethyl-2,4-cyclopentadien-1-yl]]di-,.mu.-hydroxyhexakis(pentafluorophenyl)diborate(1-), compd. with methylbenzene (2:3) (9CI) (CA INDEX NAME)

CM 1

CRN 108-88-3 CMF C7 H8

10085368.2

Page 91

CM

CRN 334023-89-1

CMF C36 H B2 F30 O . C24 H31 In2

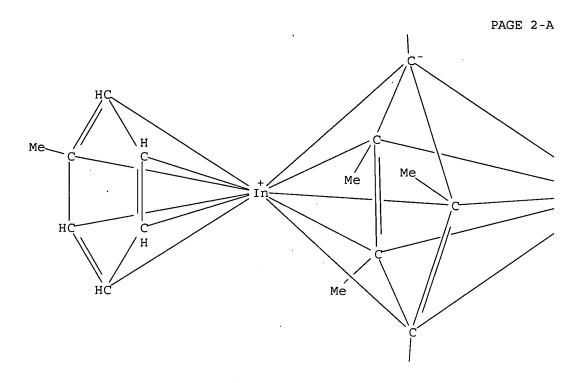
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334023-88-0 C24 H31 In2 CRN

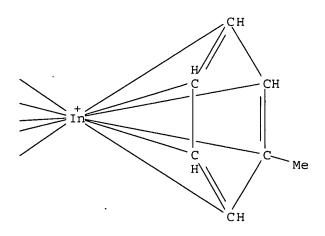
CMF

CCI CCS

PAGE 1-A



PAGE 2-B



PAGE 3-A

CM 4

CRN 219697-03-7

CMF C36 H B2 F30 O

CCI CCS

PAGE 1-A

$$F \longrightarrow F$$

PAGE 2-A

PAGE 3-A

IT 334023-89-1P

RL: PRP (Properties); SPN (Synthetic preparation); PREP (Preparation)

Patel

9/24/2003>

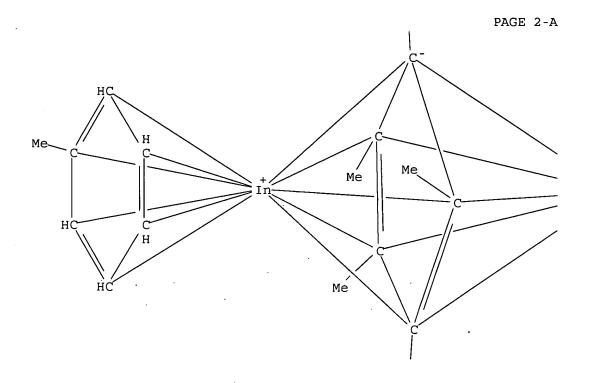
(prepn. and mol. structure of)
RN 334023-89-1 CAPLUS
CN Indium(1+), bis[(1,2,3,4,5,6-.eta.)-methylbenzene][.mu.-[(1,2,3,4,5-.eta.:1,2,3,4,5-.eta.)-1,2,3,4,5-pentamethyl-2,4-cyclopentadien-1-yl]]di-,.mu.-hydroxyhexakis(pentafluorophenyl)diborate(1-) (9CI) (CA INDEX NAME)

CM 1

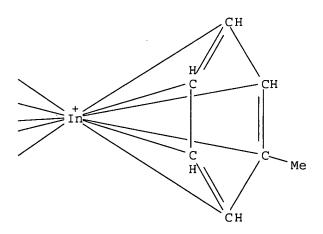
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CMF C24 H31 In2
CCI CCS

PAGE 1-A

Ме



PAGE 2-B



PAGE 3-A

CM 2

CRN 219697-03-7

CMF C36 H B2 F30 O

CCI CCS

PAGE 1-A

$$F \longrightarrow F$$

PAGE 2-A

PAGE 3-A

IT 155962-45-1, Aquatris(pentafluorophenyl)boron RL: RCT (Reactant); RACT (Reactant or reagent)

(reaction of (pentamethylcyclopentadienyl)indium with tris(pentafluorophenyl)boron in presence of toluene and)

RN 155962-45-1 CAPLUS

CN Boron, aquatris(pentafluorophenyl)-, (T-4)- (9CI) (CA INDEX NAME)

The syntheses of the first main group triple-decker cations are described, namely, [(.eta.5-C5Me5)Sn(.mu.-.eta.5-C5Me5)Sn(.eta.5-C5Me5)] [Ga(C6F5)4] and [(.eta.6-C7H8)In(.mu.-.eta.5-C5Me5)In(.eta.6-C7H8)] [(C6F5)3BO(H)B(C6F5)3], both of which have been characterized by x-ray crystallog.; the former was prepd. by the reaction of Sn(.eta.5-C5Me5)2 with Ga(C6F5)3, while the latter was prepd. by treatment of [In(.eta.5-C5Me5)] with an equimolar mixt. of B(C6F5)3 and H2O.cntdot.B(C6F5)3.

RE.CNT 21 THERE ARE 21 CITED REFERENCES AVAILABLE FOR THIS RECORD ALL CITATIONS AVAILABLE IN THE RE FORMAT

L4 ANSWER 58 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

AN 2000:887820 CAPLUS

DN 134:63915

TI Lithographic printing plate original

IN Oshima, Yasuhito; Sorori, Tadahiro

PA Fuji Photo Film Co., Ltd., Japan

SO Jpn. Kokai Tokkyo Koho, 32 pp. CODEN: JKXXAF

DT Patent

LA Japanese

FAN CNT 1

-	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
	<u>-</u>				
ΡI	JP 2000352824	A2	20001219	JP 1999-165861	19990611
				JP 1999-165861	19990611

OS MARPAT 134:63915

IT 2622-89-1, Diphenylborinic acid

RL: TEM (Technical or engineered material use); USES (Uses) (lithog. printing plate original with photosensitive layer contg. organoboronic acids)

RN 2622-89-1 CAPLUS

CN Borinic acid, diphenyl- (6CI, 7CI, 8CI, 9CI) (CA INDEX NAME)

AB A lithog. printing plate comprises a photopolymerizable photosensitive layer contg. a compd. bearing an addn.-polymerizable ethylenic unsatd. bond, a photopolymn. initiator, and a binder polymer and an intermediate layer contg. an organoboronic acid compd. sequentially fabricated on a hydrophilic support. This printing plate original possesses both high adhesiveness between imaged parts and the support and excellent stainability and is suitable for imaging using laser beam, in particular in direct plate-making by computer-to-plate (CTP) technol.

L4 ANSWER 59 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

AN 2000:875857 CAPLUS

DN 134:178638

TI .eta.2(3e)-Vinyl Complexes and One-Electron-Transfer Reactions: Tris(pentafluorophenyl)borane as a One-Electron Oxidant

AU Beddows, Claire J.; Burrows, Andrew D.; Connelly, Neil G.; Green, Michael; Lynam, Jason M.; Paget, Timothy J.

CS Department of Chemistry, University of Bath, Bath, BA2 7AY, UK

SO Organometallics (2001), 20(2), 231-233 CODEN: ORGND7; ISSN: 0276-7333

PB American Chemical Society

DT Journal

LA English

IT 326596-18-3P

RL: SPN (Synthetic preparation); PREP (Preparation) (one electron transfer/carbonylation reaction of molybdenacyclopropene complex to give)

RN 326596-18-3 CAPLUS

CN Molybdenum(1+), dicarbonyl(.eta.5-2,4-cyclopentadien-1-yl)bis(trimethyl phosphite-.kappa.P)-, stereoisomer, (T-4)-hydroxytris(pentafluorophenyl)bo rate(1-) (9CI) (CA INDEX NAME)

CM 1

CRN 148657-98-1 CMF C18 H B F15 O CCI CCS

$$F \longrightarrow C \longrightarrow B \xrightarrow{S+-} F$$

$$F \longrightarrow F$$

$$F \longrightarrow F$$

$$F \longrightarrow F$$

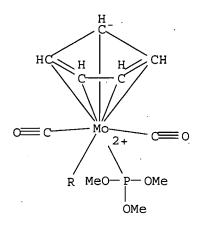
$$F \longrightarrow F$$

CM 2

CRN 135214-22-1

CMF C13 H23 Mo O8 P2

CCI CCS



AB The .eta.2(3e)-vinyl complex [Mo $\{:C(Ph)CHPh\}\{P(OMe)3\}2Cp]$ is oxidized by [FeCp2]+, [CPh3]+, or B(C6F5)3 to form the 17-electron cation [Mo $\{:C(Ph)CHPh\}\{P(OMe)3\}2Cp]+$, which on warming loses H to form the cationic .eta.2(4e)-alkyne complex [Mo(.eta.2-PhC.tplbond.CPh) $\{P(OMe)3\}2Cp]+$. In the case of the borane there is evidence for a competing reaction between the .eta.2-vinyl complex and the acid (H2O)B(C6F5)3, giving a labile trans-stilbene complex.

RE.CNT 37 THERE ARE 37 CITED REFERENCES AVAILABLE FOR THIS RECORD ALL CITATIONS AVAILABLE IN THE RE FORMAT

L4 ANSWER 60 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

AN 2000:809459 CAPLUS

DN 134:115987

TI Structural studies of organoboron compounds LXXII. Nitrones and oximes of bifunctional carbonyl compounds and their reaction products with diarylborinic acids. Crystal and molecular structure of examples of five-, six-, and seven-membered boron chelates

AU Kliegel, Wolfgang; Lubkowitz, Gottfried; Pokriefke, Jens O.; Rettig, Steven J.; Trotter, James

CS Institut fur Pharmazeutische Chemie der Technischen Universitat Braunschweig, Braunschweig, 38106, Germany

SO Canadian Journal of Chemistry (2000), 78(10), 1325-1344 CODEN: CJCHAG; ISSN: 0008-4042

PB National Research Council of Canada

DT Journal

LA English

IT 62981-91-3 89566-59-6

Page 100

RL: RCT (Reactant); RACT (Reactant or reagent)
 (reaction of carbonyl compd. nitrones and oximes with diarylborinic
 acids)

RN 62981-91-3 CAPLUS

CN Borinic acid, di-1-naphthalenyl- (9CI) (CA INDEX NAME)

RN 89566-59-6 CAPLUS

CN Borinic acid, bis(4-chlorophenyl) - (9CI) (CA INDEX NAME)

IT 73774-45-5P

RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)

(reaction of carbonyl compd. nitrones and oximes with diarylborinic acids)

RN 73774-45-5 CAPLUS

CN Borinic acid, bis(4-methoxyphenyl) - (9CI) (CA INDEX NAME)

GI

AΒ Synthesis has been carried out of diarylboron chelates of 2- and 3-hydroxynitrones, of 2- and 3-hydroxyoximes, and of 2-carboxynitrones and a 2-carboxyoxime. The structures have been detd. from spectroscopic data and from x-ray analyses of boron chelates I (5d, R = Me3C, R1 = H, R2 =Me), II, III, and I (19, R = Ph, R1R2 = O). Crystals (at 180 K) of 5d are monoclinic, a = 10.543(2), b = 19.085(4), c = 10.2667(3) .ANG., .beta. = 90.4978(7).degree., Z = 4, space group P21/c; those of II areorthorhombic, a = 10.9913(5), b = 14.9329(7), c = 10.2460(13) .ANG., Z = 10.2460(13)4, space group P212121; those of III are monoclinic, a = 11.227(2), b = 9.967(2), c = 17.0537(4) .ANG., .beta. = 105.4179(5).degree., Z = 4, space group P21/n; those of 19 are monoclinic, a = 11.1847(15), b = 13.715(3), c = 11.5559(5) .ANG., .beta. = 104.8730(10).degree., Z = 4, space group P21/n. The structures were solved by direct methods and refined by full-matrix least-squares procedures to R(F, I .gtoreq. 3.sigma.(I)) =0.049, 0.047, 0.042, and 0.047, resp., for CCD data for 5d, II, III, and 19. The four mols. contain five-, seven-, six-, and five-membered rings, resp., with O-B-N groups in the 5d, III, and 19, and O-B-O in II; the rings exhibit various deviations from planarity, particularly the seven-membered ring.

RE.CNT 77 THERE ARE 77 CITED REFERENCES AVAILABLE FOR THIS RECORD ALL CITATIONS AVAILABLE IN THE RE FORMAT

L4 ANSWER 61 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

AN 2000:808127 CAPLUS

DN 134:115992

TI Diverse Reactivity of Dialkylaluminum Dimesitylboryloxides [(.mu.-Mes2BO)AlR2]2. Synthetic and Structural Study

AU Anulewicz-Ostrowska, Romana; Lulinski, Sergiusz; Serwatowski, Janusz; Suwinska, Kinga

CS Faculty of Chemistry, University of Warsaw, Warsaw, 02-093, Pol.

SO Inorganic Chemistry (2000), 39(25), 5763-5767 CODEN: INOCAJ; ISSN: 0020-1669

PB American Chemical Society

DT Journal

LA English

OS CASREACT 134:115992

IT 20631-84-9, Dimesitylborinic acid

RL: RCT (Reactant); RACT (Reactant or reagent)

(catalytic decompn. and reaction with trialkylaluminums)

RN 20631-84-9 CAPLUS

CN Borinic acid, bis(2,4,6-trimethylphenyl) - (9CI) (CA INDEX NAME)

AB Several stable dimeric dialkylaluminum boryloxides of the formula [(.mu.-Mes2BO)AlR2]2 (R = Me (1), Et (2), iBu) have been prepd. from dimesitylborinic acid Mes2BOH and trialkylaluminums R3Al. Compd. 1 has been characterized by x-ray diffraction. These compds. exhibit diverse reactivity toward protonolytic reagents depending on the bulkiness of these reagents. Treatment of 1 with tert-Bu alc. afforded cryst. species trans-[(.mu.-tBuO)(Mes2BO)AlMe]2 (3), which is the first example of a mixed system contg. boryloxide and alkoxide ligands together. Most surprisingly, Mes2BOH was found to undergo catalytic decompn. in the presence of [(.mu.-Mes2BO)AlR2]2 via the unexpected cleavage of one boron-carbon bond. The mol. structure of the decompn. product, i.e., trimesitylboroxin [MesBO]3 (4) is reported.

RE.CNT 30 THERE ARE 30 CITED REFERENCES AVAILABLE FOR THIS RECORD ALL CITATIONS AVAILABLE IN THE RE FORMAT

L4 ANSWER 62 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

AN 2000:734616 CAPLUS

DN 134:42174

TI Aqua, Alcohol, and Acetonitrile Adducts of Tris(perfluorophenyl)borane: Evaluation of Bronsted Acidity and Ligand Lability with Experimental and Computational Methods

AU Bergquist, Catherine; Bridgewater, Brian M.; Harlan, C. Jeff; Norton, Jack R.; Friesner, Richard A.; Parkin, Gerard

CS Department of Chemistry, Columbia University, New York, NY, 10027, USA

SO Journal of the American Chemical Society (2000), 122(43), 10581-10590 CODEN: JACSAT; ISSN: 0002-7863

PB American Chemical Society

DT Journal

LA English

IT 148657-98-1

RL: FMU (Formation, unclassified); PEP (Physical, engineering or chemical process); PRP (Properties); RCT (Reactant); FORM (Formation, nonpreparative); PROC (Process); RACT (Reactant or reagent)

(aqua, alc., and acetonitrile adducts of tris(perfluorophenyl)borane and evaluation of Bronsted acidity and ligand lability with exptl. and computational methods)

RN 148657-98-1 CAPLUS

CN Borate(1-), hydroxytris(pentafluorophenyl)-, (T-4)- (9CI) (CA INDEX NAME)

$$F \xrightarrow{F} G \xrightarrow{OH^{-}} F \xrightarrow{F} F$$

$$F \xrightarrow{F} F \xrightarrow{F} F$$

IT 312640-07-6 312640-08-7

RL: FMU (Formation, unclassified); PRP (Properties); FORM (Formation, nonpreparative)

(aqua, alc., and acetonitrile adducts of tris(perfluorophenyl)borane and evaluation of Bronsted acidity and ligand lability with exptl. and computational methods)

RN 312640-07-6 CAPLUS

CN Boron, aquatris(pentafluorophenyl)-, (T-4)-, compd. with acetonitrile (1:1) (9CI) (CA INDEX NAME)

CM 1

CRN 155962-45-1 CMF C18 H2 B F15 O

CCI CCS

CM 2

CRN 75-05-8 CMF C2 H3 N

H3C-C≡N

RN 312640-08-7 CAPLUS

CN Boron, aquatris(pentafluorophenyl)-, (T-4)-, compd. with acetonitrile (1:1), monohydrate (9CI) (CA INDEX NAME)

CM 1

CRN 155962-45-1 CMF C18 H2 B F15 O

CCI CCS

$$F \longrightarrow C \longrightarrow B \xrightarrow{S+-} F$$

$$F \longrightarrow F$$

$$F \longrightarrow F$$

$$F \longrightarrow F$$

$$F \longrightarrow F$$

CM2

CRN 75-05-8 C2 H3 N CMF

 $H_3C-C = N$

ΙT 155962-44-0 155962-46-2 312640-05-4 312640-06-5

RL: FMU (Formation, unclassified); PRP (Properties); FORM (Formation,

nonpreparative)

(mol. structure calcn.; aqua, alc., and acetonitrile adducts of tris(perfluorophenyl)borane and evaluation of Bronsted acidity and ligand lability with exptl. and computational methods)

RN155962-44-0 CAPLUS

Boron, aquatris(pentafluorophenyl)-, dihydrate, (T-4)- (9CI) CN(CA INDEX NAME)

$$F \longrightarrow F \longrightarrow F \longrightarrow F \longrightarrow F$$

●2 H₂O

RN 155962-46-2 CAPLUS

CN Boron, (methanol)tris(pentafluorophenyl)-, (T-4)- (9CI) (CA INDEX NAME)

RN 312640-05-4 CAPLUS

CN Boron, aquatris(pentafluorophenyl)-, monohydrate, (T-4)- (9CI) (CA INDEX NAME)

$$F \longrightarrow F \longrightarrow F$$

$$F \longrightarrow F$$

H₂O

RN 312640-06-5 CAPLUS

CN Boron, (2-methyl-2-propanol)tris(pentafluorophenyl)-, (T-4)- (9CI) (CA INDEX NAME)

$$F \longrightarrow F$$

$$R \longrightarrow C \longrightarrow F$$

IT 155962-45-1P 155962-47-3P 312640-04-3P

RL: PEP (Physical, engineering or chemical process); PRP (Properties); SPN (Synthetic preparation); PREP (Preparation); PROC (Process) (prepn. and crystal and mol. structure of)

RN 155962-45-1 CAPLUS

CN Boron, aquatris(pentafluorophenyl)-, (T-4)- (9CI) (CA INDEX NAME)

RN 155962-47-3 CAPLUS

CN Boron, (methanol)tris(pentafluorophenyl)-, (T-4)-, compd. with methanol (1:1) (9CI) (CA INDEX NAME)

CM 1

CRN 155962-46-2

CMF C19 H4 B F15 O CCI CCS

CM 2

CRN 67-56-1 CMF C H4 O

нзс-он

RN 312640-04-3 CAPLUS

CN Boron, aquatris(pentafluorophenyl)-, (T-4)-, compd. with 2-methyl-2-propanol (1:1) (9CI) (CA INDEX NAME)

CM 1

CRN 155962-45-1 CMF C18 H2 B F15 O CCI CCS

$$F \xrightarrow{F} C \xrightarrow{OH_2} F \xrightarrow{F} F$$

$$F \xrightarrow{F} F \xrightarrow{F} F$$

CM 2

CRN 75-65-0 CMF C4 H10 O

IT 312640-09-8P

RN 312640-09-8 CAPLUS

CN Borate(1-), hydroxytris(pentafluorophenyl)-, (T-4)-, hydrogen, compd. with N,N,N',N'-tetramethyl-1,8-naphthalenediamine (1:1) (9CI) (CA INDEX NAME)

CM 1

CRN 147892-17-9 CMF C18 H B F15 O . H CCI CCS

$$F \longrightarrow F \longrightarrow F \longrightarrow F \longrightarrow F$$

● H+

CM 2

CRN 20734-58-1 CMF C14 H18 N2

AB Equil. studies have been performed to det. the Bronsted acidity of [(C6F5)3B(OH2)].H2O, the aqua species that exists in acetonitrile solns.

of B(C6F5)3 in the presence of water. NMR spectroscopic anal. of the deprotonation of [(C6F5)3B(OH2)].H2O with 2,6-But2C5H3N in acetonitrile allows a pK value of 8.6 to be detd. for the equil. [(C6F5)3B(OH2)].H2O. dblarw. [(C6F5)3B(OH)] - + [H3O] +. On the basis of a calcd. value for the hydrogen bond interaction in [(C6F5)3B(OH2)].H2O, the pKa for (C6F5)3B(OH2) is estd. to be 8.4 in acetonitrile. Such a value indicates that (C6F5)3B(OH2) must be regarded as a strong acid, with a strength comparable to that of HCl in acetonitrile. Dynamic NMR spectroscopic studies indicate that the aqua and acetonitrile ligands in (C6F5)3B(OH2) and (C6F5)3B(NCMe) are labile, with dissocn. of H2O being substantially more facile than that of MeCN, by a factor of ca. 200 in rate const. at 300 K. Ab initio calcns. were performed in the gas phase and with a dielec. solvent model to det. the strength of B-L bonds (L = H2O), ROH, MeCN) and hydrogen bonds involving B-OH2 and B-O(H)R derivs.

RE CNT 60 THERE ARE 60 CITED REFERENCES AVAILABLE FOR THIS RECORD ALL CITATIONS AVAILABLE IN THE RE FORMAT

- L4 ANSWER 63 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN
- AN 2000:732648 CAPLUS
- DN 134:43364
- TI Synthesis and properties of fluorescent organoboranes: triarylmethane-type dyes
- AU Albrecht, Karsten; Kaiser, Volker; Boese, Roland; Adams, Jorg; Kaufmann, Dieter E.
- CS Institut fur Organische Chemie, Technische Universitat Clausthal, Clausthal-Zellerfeld, 38678, Germany
- SO Perkin 2 (2000), (10), 2153-2157
- CODEN: PRKTFO; ISSN: 1470-1820 PB Royal Society of Chemistry
- DT Journal
- LA English
- OS CASREACT 134:43364
- IT 313220-06-3P

RL: PRP (Properties); SPN (Synthetic preparation); TEM (Technical or engineered material use); PREP (Preparation); USES (Uses) (dye; synthesis and properties of fluorescent organoborane

triarylmethane-type dyes)

- RN 313220-06-3 CAPLUS
- CN Borinic acid, [2-(diethylamino)phenyl](2,3,5,6-tetramethylphenyl)- (9CI) (CA INDEX NAME)

AB The syntheses and photochem. properties of the novel aminoaryldiarylboranes which are isoelectronic with triarylmethane dyes, and also pyrrolyl- and indolyldiarylboranes, are described. The fluorescence spectra are strongly dependent on the solvent. The use of o-disubstituted arenes as stabilizing substituents at the boron atom leads to highly colored solids which are stable to air and moisture. The

structures of two of the triarylboranes were confirmed by X-ray analyses.
RE.CNT 37 THERE ARE 37 CITED REFERENCES AVAILABLE FOR THIS RECORD
ALL CITATIONS AVAILABLE IN THE RE FORMAT

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ANSWER 64 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN
L4
     2000:441802 CAPLUS
AN
DN
     133:74149
TI
     Method for producing mono- or di-organoboranes
IN
     Schottek, Jorg; Becker, Patricia; Kullmer, Iris
PA
     Targor G.m.b.H., Germany
SO
     PCT Int. Appl., 19 pp.
     CODEN: PIXXD2
DT
     Patent
     German
LΑ
FAN.CNT 1
     PATENT NO.
                      KIND DATE
                                           APPLICATION NO.
                            -----
                                           _____
PΙ
     WO 2000037476
                      A1
                            20000629
                                           WO 1999-EP10032 19991217
         W: BR, CN, JP, NO, US
         RW: AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL,
             PT, SE
                                           DE 1998-19858829A 19981219
     BR 9916382
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                            20010911
                                           BR 1999-16382
                                                            19991217
                                           DE 1998-19858829A 19981219
                                           WO 1999-EP10032W 19991217
     EP 1140947
                       Α1
                            20011010
                                           EP 1999-963568
                                                            19991217
     EP 1140947
                       В1
                            20030312
         R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT,
             IE, FI
                                           DE 1998-19858829A 19981219
                                           WO 1999-EP10032W 19991217
     JP 2002533349
                       T2
                            20021008
                                           JP 2000-589546
                                                            19991217
                                           DE 1998-19858829A 19981219
                                           WO 1999-EP10032W 19991217
    US 6600066
                       В1
                            20030729
                                           US 2001-868074
                                                            20010614
                                           DE 1998-19858829A 19981219
                                           WO 1999-EP10032W 19991217
     NO 2001003021
                            20010809
                                           NO 2001-3021
                                                             20010618
                                           DE 1998-19858829A 19981219
                                           WO 1999-EP10032W 19991217
OS
     CASREACT 133:74149; MARPAT 133:74149
IT
     2118-02-7P, Hydroxybis (pentafluorophenyl) borane 2622-89-1P
     73774-44-4P 73774-45-5P 89566-59-6P
     211636-23-6P 279216-30-7P 279216-31-8P
     RL: SPN (Synthetic preparation); PREP (Preparation)
        (prepn. of)
     2118-02-7 CAPLUS
RN
CN
     Borinic acid, bis(pentafluorophenyl) - (7CI, 8CI, 9CI) (CA INDEX NAME)
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$$F \longrightarrow F \longrightarrow F$$

RN 2622-89-1 CAPLUS

CN Borinic acid, diphenyl- (6CI, 7CI, 8CI, 9CI) (CA INDEX NAME)

RN 73774-44-4 CAPLUS

CN Borinic acid, bis(2-methylphenyl) - (9CI) (CA INDEX NAME)

RN 73774-45-5 CAPLUS

CN Borinic acid, bis(4-methoxyphenyl)- (9CI) (CA INDEX NAME)

RN 89566-59-6 CAPLUS

CN Borinic acid, bis(4-chlorophenyl) - (9CI) (CA INDEX NAME)

RN 211636-23-6 CAPLUS

CN Borinic acid, bis[3,5-bis(trifluoromethyl)phenyl] - (9CI) (CA INDEX NAME)

RN 279216-30-7 CAPLUS

CN Borinic acid, bis(2',3',4',5',6'-pentafluoro[1,1'-biphenyl]-4-yl)- (9CI) (CA INDEX NAME)

RN 279216-31-8 CAPLUS

CN Borinic acid, bis([1,1'-biphenyl]-4-yl)- (9CI) (CA INDEX NAME)

AB The invention relates to a novel method for producing perhalogenated mono-organoboranes or di-organoboranes which makes it possible to obtain these compds. under conditions which can be carried out in a tech. favorable manner. Thus, reaction of tris(pentafluorophenyl)borane with H2O in toluene gave 89% bis(pentafluorophenyl)hydroxyborane.

RE.CNT 2 THERE ARE 2 CITED REFERENCES AVAILABLE FOR THIS RECORD
ALL CITATIONS AVAILABLE IN THE RE FORMAT

L4 ANSWER 65 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

AN 2000:401797 CAPLUS

DN 133:43531

TI Process for preparing pyridazin-3-one derivatives

IN Yanagawa, Masao; Mizuno, Masahiko; Oda, Yoshiaki

PA Sumitomo Chemical Company, Limited, Japan

SO PCT Int. Appl., 38 pp.

CODEN: PIXXD2

DT Patent

LA English

FAN. CNT 1

PATENT NO. KIND DATE APPLICATION NO. DATE
PI WO 2000034249 A1 20000615 WO 1999-JP6842 19991207

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10085368.2
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Page 113

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AE, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CR, CU,
        CZ, DE, DK, DM, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL,
         IN, IS, KE, KG, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG,
        MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL,
        TJ, TM, TR, TT, TZ, UA, UG, US, UZ, VN, YU, ZA, ZW, AM, AZ, BY,
        KG, KZ, MD, RU, TJ, TM
    RW: GH, GM, KE, LS, MW, SD, SL, SZ, TZ, UG, ZW, AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, BF, BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG
                                         JP 1998-350274 A 19981209
                                         JP 1999-172464 A 19990618
                         20000822
JP 2000229948
                   A2
                                         JP 1999-172464
                                                            19990618
                                         JP 1998-350274 A 19981209
AU 2000015128
                         20000626
                                         AU 2000-15128
                   A5
                                                            19991207
                                          JP 1998-350274 A 19981209
                                          JP 1999-172464 A 19990618
                                         WO 1999-JP6842 W 19991207
BR 9916028
                         20010828
                                         BR 1999-16028
                                                            19991207
                                         JP 1998-350274 A 19981209
                                         JP 1999-172464 A 19990618
                                         WO 1999-JP6842 W 19991207
                   A1.
EP 1137641
                         20011004
                                         EP 1999-957420
                                                           19991207
    R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT,
        IE, SI, LT, LV, FI, RO
                                         JP 1998-350274 A 19981209
                                         JP 1999-172464 A 19990618
                                         WO 1999-JP6842 W 19991207
US 6500951
                   В1
                         20021231
                                         US 2001-857033
                                                            20010628
                                         JP 1998-350274 A 19981209
                                         JP 1999-172464 A 19990618
                                         WO 1999-JP6842 W 19991207
MARPAT 133:43531
2622-89-1, Diphenylborinic acid
RL: RCT (Reactant); RACT (Reactant or reagent)
   (cyclization of 3-carboxy-2-hydroxy-2-(trifluoromethyl)butanal
   (4-chloro-2-fluoro-5-hydroxyphenyl)hydrazone in presence of)
2622-89-1 CAPLUS
Borinic acid, diphenyl- (6CI, 7CI, 8CI, 9CI) (CA INDEX NAME)
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GI

OS

ΙT

RN

10085368.2

Page 114

AB Pyridazin-3-ones [I; R2 = H, C1-C3 alkyl; R3 = H, C1-C3 alkyl; Q = (un)substituted phenyl] are prepd. by cyclization of a carboxylic acid deriv. (II; same R2, R3, Q) in the presence of a nitrogen-contg. arom. compd. and a boron compd. Thus, 5.62 g II (R2 = Me, R3 = H, Q = 4-chloro-2-fluoro-5-hydroxyphenyl), 0.38 g phenylboric acid, 2.70 g mol. sieve 4A, and 2.82 g 5-ethyl-2-methylpyridine were stirred at 110-115.degree. for 10 h to give a 91.2% yield of I (R2 = Me, R3 = H, Q = 4-chloro-2-fluoro-5-hydroxyphenyl).

RE CNT 4 THERE ARE 4 CITED REFERENCES AVAILABLE FOR THIS RECORD ALL CITATIONS AVAILABLE IN THE RE FORMAT

L4 ANSWER 66 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

AN 2000:253333 CAPLUS

DN 133:58910

TI Para-Substituted diphenylborylated organocobaloximes: effects of substituents on conformation and redox properties

AU Asaro, Fioretta; Dreos, Renata; Nardin, Giorgio; Pellizer, Giorgio; Peressini, Silvia; Randaccio, Lucio; Siega, Patrizia; Tauzher, Giovanni; Tavagnacco, Claudio

CS Dipartimento di Scienze Chimiche, Universita Degli Studi di Trieste, Trieste, I-34127, Italy

SO Journal of Organometallic Chemistry (2000), 601(1), 114-125 CODEN: JORCAI; ISSN: 0022-328X

PB Elsevier Science S.A.

DT Journal

LA English

OS CASREACT 133:58910

IT 2622-89-1 66117-64-4 73774-45-5 89566-59-6

RL: RCT (Reactant); RACT (Reactant or reagent)
 (reactions with cobaloximes)

RN 2622-89-1 CAPLUS

CN Borinic acid, diphenyl- (6CI, 7CI, 8CI, 9CI) (CA INDEX NAME)

RN 66117-64-4 CAPLUS

CN Borinic acid, bis(4-methylphenyl) - (9CI) (CA INDEX NAME)

RN 73774-45-5 CAPLUS

CN Borinic acid, bis(4-methoxyphenyl) - (9CI) (CA INDEX NAME)

RN 89566-59-6 CAPLUS
CN Borinic acid, bis(4-chlorophenyl)- (9CI) (CA INDEX NAME)

GI

Ι

AB Some new derivs. of organocobaloximes contg. para-substituted diphenylboryl groups, RCo(DH)2-n(DB(p-XPh)2)nL (R = alkyl or aryl group, L = N-MeIm, Py or H2O, X = OCH3, CH3 or Cl, n = 1 or 2) (shown as I for n = 1) were synthesized. The x-ray structures and the 1H-NMR spectra are compared with those of the corresponding RCo(DH)2-n(DBPh2)nL and RCo(DBF2)2L complexes. The insertion of X groups in the Ph rings does not significantly affect the equatorial Co-N distances, whereas the Co-Py distances increase slightly in the order (DB(p-OCH3Ph)2)2<(DB(p-ClPh)2)2<(DBF2)2. '1H-NMR spectra suggest that the conformational distribution in soln. is similar to that obsd. in the corresponding BPh2 derivs. Electrochem. studies on the corresponding MeCo(DB(p-XPh)2)2H2O compds. show a mono-electron Co(III)/Co(II) transfer reaction followed by two parallel reactions: (a) mono-electron Co(III)/Co(I) transfer; (b)

9/24/2003>

homolytic dissocn. of the Co-C bond with the formation of Co(I) species, the relative rates of the two processes being dependent on X. As the electron-withdrawing power of the equatorial ligand increases, the redn. potentials assocd. with both Co(III)/Co(II) and Co(III)/Co(I) processes shift toward pos. values, indicating a decrease of electron d. on the Co atom. The effects are comparable with those obsd. by changing the axial ligands.

RE.CNT 35 THERE ARE 35 CITED REFERENCES AVAILABLE FOR THIS RECORD ALL CITATIONS AVAILABLE IN THE RE FORMAT

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ANSWER 67 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN
L4
AN
    2000:241304 CAPLUS
DN
    132:279652
TΙ
    Catalysts for the polymerization of olefins
IN
    Schottek, Jorg; Fritze, Cornelia; Bohnen, Hans; Becker, Patricia
PA
    Targor Gmbh, Germany
SO
    PCT Int. Appl., 56 pp.
    CODEN: PIXXD2
DT
    Patent
LΑ
    German
FAN.CNT 5
    PATENT NO. KIND DATE
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    WO 2000020466 A1
                         20000413 WO 1999-EP7087 19990923
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DE 19903306 A1
                         20000803
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                                     APPLICATION NO. DATE
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FAN 2000:396574
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                  KIND DATE
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                   A1
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    WO 2000035973
                    A1
                         20000622
                                      WO 1999-EP9682 19991209
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                                      DE 1999-19903306A 19990128
    EP 1054914
                    A1 20001129
                                      EP 1999-962247 19991209
       R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT,
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                                      DE 1998-19857377A 19981212
                                      DE 1999-19903306A 19990128
                                      WO 1999-EP9682 W 19991209
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	JP 2002532584	T2 20021002	JP 2000-588228 19991209 DE 1998-19857377A 19981212 DE 1999-19903306A 19990128 WO 1999-EP9682 W 19991209
	US 6486277	B1 20021126	
FAN		KIND DATE	APPLICATION NO. DATE
DI	WO 200025072	71 20000622	HO 1000 BB0602 10001000
ΡΙ	W: BR, JP,	US	WO 1999-EP9682 19991209
	RW: AT, BE, PT, SE	CH, CY, DE, DK,	ES, FI, FR, GB, GR, IE, IT, LU, MC, NL,
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	DE 19857377	A1 20000615	DE 1998-19857377 19981212
	DE 19903306	A1 20000803	DE 1999-19903306 19990128
			EP 1999-962247 19991209
	R: AT, BE, IE, FI	CH, DE, DK, ES,	FR, GB, GR, IT, LI, LU, NL, SE, MC, PT,
	12, 11		DE 1998-19857377A 19981212
	• •		DE 1999-19903306A 19990128
	TD 2002522504	ma 20021002	WO 1999-EP9682 W 19991209
	JP 2002532584	T2 20021002	JP 2000-588228 19991209 DE 1998-19857377A 19981212
			DE 1999-19903306A 19990128
			WO 1999-EP9682 W 19991209
	US 6486277	B1 20021126	
			DE 1998-19857377A 19981212 DE 1999-19903306A 19990128
			WO 1999-EP9682 W 19991209
FAN	2000:535190 PATENT NO.	KIND DATE	APPLICATION NO. DATE
5.7			
ΡΙ	WO 2000044799 W: BR, JP,		WO 2000-EP471 20000122
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	DE 19903306 BR 2000004493	A1 20000803	
	BR 2000004493	A 20001219	BR 2000-4493 20000122 DE 1999-19903306A 19990128
			WO 2000-EP471 W 20000122
	EP 1082363		EP 2000-910601 20000122
	R: AT, BE, IE, FI	CH, DE, DK, ES,	FR, GB, GR, IT, LI, LU, NL, SE, MC, PT,
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	JP 2002535416	T2 20021022	WO 2000-EP471 W 20000122 JP 2000-596054 20000122
	UF 2002333410	12 20021022	DE 1999-19903306A 19990128
			WO 2000-EP471 W 20000122
	US 6469114	B1 20021022	
			DE 1999-19903306A 19990128 WO 2000-EP471 W 20000122
os	MARPAT 132:2796	52	110 2000 BL471 W 20000122

OS MARPAT 132:279652
IT 2118-02-7, Bis(pentafluorophenyl)borinic acid

Patel 9/24/2003>

RL: RCT (Reactant); RACT (Reactant or reagent) (dehydration)

RN 2118-02-7 CAPLUS

CN Borinic acid, bis(pentafluorophenyl) - (7CI, 8CI, 9CI) (CA INDEX NAME)

AB The title catalysts, having high activity and giving polymers with good morphol., contain metallocenes, Lewis bases of specified structure based on Group VA elements (esp. N or P), supports, and org. B or Al compds. of specified structure. Heating (C6F5)2BOH at 100.degree. in vacuo for 8 h gave 92% corresponding anhydride which was mixed (20.8 mmol) with 20.8 mmol PhNMe2 and 14.0 g SiO2 in PhMe and dried in vacuo to give a cocatalyst. Mixing the cocatalyst (996 mg) with 5 mg [(dimethylsilylene)bis(2-methyl-4-phenylindenyl)]zirconium dichloride ans 70 .mu.mol Me3Al in PhMe and drying in vacuo gave 1.001 g free-flowing, powd. catalyst. Heating 1 g catalyst with 0.5 mL 20% iso-Bu3Al and 10 dm3 liq. C3H6 at 60.degree. for 1 h gave 485 g polypropylene (97 kg/g metallocene-h).

RE.CNT 6 THERE ARE 6 CITED REFERENCES AVAILABLE FOR THIS RECORD ALL CITATIONS AVAILABLE IN THE RE FORMAT

ANSWER 68 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN L4

ΑN 2000:227941 CAPLUS

DN 132:251567

TΙ Metallocene-based olefin polymerization catalyst systems and their use

IN Schottek, Joerg; Fritze, Cornelia; Bohnen, Hans; Becker, Patricia

PΑ Aventis Research & Technologies G.m.b.H. & Co. K.-G., Germany

SO Ger. Offen., 11 pp.

CODEN: GWXXBX

DT Patent

LA German

FAN.CNT 5

	PATENT NO.	KIND	DATE	. APPLICATION NO.	DATE
ΡI	DE 19845240	A1	20000406	DE 1998-19845240	19981001
	WO 2000020466	A1	20000413	WO 1999-EP7087	19990923
	W: JP, KR,	US			
	RW: AT, BE,	CH, CY	, DE, DK, ES,	FI, FR, GB, GR, IE,	, IT, LU, MC, NL,
•	PT, SE				

DE 1998-19845240A 19981001 DE 1999-19903306A 19990128

PATENT FAMILY INFORMATION:

2000:241304

PATENT NO. KIND DATE APPLICATION NO. DATE PΙ WO 2000020466 A1 20000413 WO 1999-EP7087 19990923 W: JP, KR, US

RW: AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL,

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	11, 55				DE 1998-19845240A 19981001	
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FAN	2000:396574					
	PATENT NO.	KIND	DATE		APPLICATION NO. DATE	
ΡI	DE 19857377		20000615		DE 1998-19857377 19981212	
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	IE, FI				DE 1000 100572777 10001212	
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	TD 0000530504	 0			WO 1999-EP9682 W 19991209	
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			•		DE 1999-19903306A 19990128	
	US 6486277	Вl	20021126		WO 1999-EP9682 W 19991209 US 2000-622205 20000814	
	05 0100277	DI	20021120		DE 1998-19857377A 19981212	
					DE 1999-19903306A 19990128 WO 1999-EP9682 W 19991209	
FAN	2000:421191					
FAN	PATENT NO.				APPLICATION NO. DATE	
FAN PI			DATE 20000622			
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	PATENT NO. WO 2000035973 W: BR, JP, RW: AT, BE, PT, SE DE 19857377 DE 19903306	A1 US CH, CY	20000622 , DE, DK, 20000615 20000803	ES,	APPLICATION NO. DATE WO 1999-EP9682 19991209 FI, FR, GB, GR, IE, IT, LU, MC, NI DE 1998-19857377A 19981212 DE 1999-19903306A 19990128 DE 1998-19857377 19981212 DE 1999-19903306 19990128	٠,
	PATENT NO. WO 2000035973 W: BR, JP, RW: AT, BE, PT, SE DE 19857377 DE 19903306 EP 1054914 R: AT, BE,	A1 US CH, CY A1 A1 A1	20000622 , DE, DK, 20000615 20000803 20001129	ES,	APPLICATION NO. DATE WO 1999-EP9682 19991209 FI, FR, GB, GR, IE, IT, LU, MC, NI DE 1998-19857377A 19981212	
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	PATENT NO. WO 2000035973 W: BR, JP, RW: AT, BE, PT, SE DE 19857377 DE 19903306 EP 1054914 R: AT, BE, IE, FI	A1 US CH, CY A1 A1 A1 CH, DE	20000622 , DE, DK, 20000615 20000803 20001129 , DK, ES,	ES,	APPLICATION NO. DATE	
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OS

2118-02-7, Bis (pentafluorophenyl) borinic acid ITRL: RCT (Reactant); RACT (Reactant or reagent)

(prepn. of metallocene-based olefin polymn. catalyst systems)

RN 2118-02-7 CAPLUS

Borinic acid, bis(pentafluorophenyl) - (7CI, 8CI, 9CI) (CA INDEX NAME) CN

AB The catalyst system consists of a metallocene, a Lewis base, a carrier, an organoboron or organoaluminum compd., and optionally a further organometallic compd. A high catalyst activity and a good polymer morphol. are obtained without the use of aluminoxanes as cocatalysts. Thus, (C6F5) 2BOH was heated 8 h at 100.degree. under high vacuum to produce (C6F5)2BOB(C6F5)2, which was deposited on SiO2 pretreated with PhNMe2 to give a supported cocatalyst. A toluene soln. of [(dimethylsilylene)bis(2-methyl-4-phenylindenyl)]zirconium dichloride was mixed with Me3Al and then with the supported cocatalyst, and the solvent was evapd. to give a catalyst powder. The activity of the catalyst powder for bulk polymn. of propylene at 60.degree. was 97 kg polymer/g metallocene per h.

- ANSWER 69 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN L4
- 2000:188361 CAPLUS ΑN
- DN 132:194495
- Procedures for production of mono or Di-organo-boranes ΤI
- IN Bohnen, Hans; Hahn, Ulrich
- Aventis Research & Technologies G.m.b.H. & Co. K.-G., Germany PΑ
- SO Ger. Offen., 6 pp. CODEN: GWXXBX

9/24/2003> Patel

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DT
     Patent
LΑ
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FAN.CNT 1
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                               DATE
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              KG, KZ, MD, RU, TJ, TM
          RW: GH, GM, KE, LS, MW, SD, SL, SZ, TZ, UG, ZW, AT, BE, CH, CY, DE,
              DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, BF, BJ, CF,
              CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG
                                                DE 1998-19843055A 19980919
     AU 9959788
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                                                AU 1999-59788
                                                                   19990916
                                                DE 1998-19843055A 19980919
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          R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT,
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     JP 2002526503
                         T2
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                                                JP 2000-574117
                                                                   19990916
                                                DE 1998-19843055A 19980919
                                                WO 1999-EP6861 W 19990916
OS
     CASREACT 132:194495
ΙT
     2118-02-7P, Hydroxybis (pentafluorophenyl) borane
     RL: SPN (Synthetic preparation); PREP (Preparation)
         (prepn. of)
RN
     2118-02-7 CAPLUS
CN
     Borinic acid, bis(pentafluorophenyl) - (7CI, 8CI, 9CI) (CA INDEX NAME)
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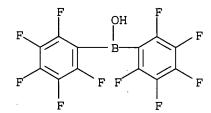
The available invention concerns a new procedure for prodn. of perhalogenated diorgano hydroxy boranes under tech. feasible conditions. Thus, Grignard arylation of Me2SnBr2 with 1-bromo-2,3,4,5,6-pentafluorobenzene in Et2O in the presence of Mg gave 93% dimethylbis(pentafluorophenyl)stannane. Reaction of dimethylbis(pentafluorophenyl)stannane with BCl3 gave 70% chlorobis(pentafluorophenyl)borane which on hydrolysis gave 96% hydroxybis(pentafluorophenyl)borane.

L4 ANSWER 70 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

AN 2000:178812 CAPLUS

DN 132:321889

- TI (Fluoroorgano) fluoroboranes and -fluoroborates. I. Synthesis and spectroscopic characterization of potassium fluoroaryltrifluoroborates and fluoroaryldifluoroboranes
- AU Frohn, H.-J.; Franke, H.; Fritzen, P.; Bardin, V. V.
- CS Fachgebiet Anorganische Chemie, Gerhard-Mercator-Universitat Duisburg, Duisburg, D-47048, Germany
- SO Journal of Organometallic Chemistry (2000), 598(1), 127-135 CODEN: JORCAI; ISSN: 0022-328X
- PB Elsevier Science S.A.
- DT Journal
- LA English
- OS CASREACT 132:321889
- IT 2118-02-7P, Bis(pentafluorophenyl)borinic acid
 RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT
 (Reactant or reagent)
 - (prepn. and reaction with potassium hydrogen difluoride)
- RN 2118-02-7 CAPLUS
- CN Borinic acid, bis(pentafluorophenyl) (7CI, 8CI, 9CI) (CA INDEX NAME)



- AB A convenient prepn. of K[ArBF3] (Ar = 2-C6H4F, 3-C6H4F, 4-C6H4F, 2,6-C6H3F2, 3,5-C6H3F2, 2,4,6-C6H2F3, 3,4,5-C6H2F3, 2,3,4,5-C6HF4, C6F5) is offered and the IR and multinuclear NMR spectra of these salts are reported. Treatment of the trifluoroborate salts with BF3 in chlorocarbon solvents provides an easy synthetic route to the corresponding aryldifluoroboranes ArBF2. The multinuclear NMR spectra of ArBF2 are presented. The electron substituent effect of the [-BF3]--group shows this substituent as one of the strongest .sigma.-electron donors, while its .pi.-electron influence is negligible (.sigma.I = -0.32, .sigma.R = -0.07).
- RE.CNT 20 THERE ARE 20 CITED REFERENCES AVAILABLE FOR THIS RECORD ALL CITATIONS AVAILABLE IN THE RE FORMAT
- L4 ANSWER 71 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN
- AN 2000:173550 CAPLUS
- DN. 132:322176
- TI Mechanistic Studies of Alkene/CO Polymerization with Palladium Complexes Promoted by B(C6F5)3
- AU Barlow, Graham K.; Boyle, Jane D.; Cooley, Neil A.; Ghaffar, Talit; Wass, Duncan F.
- CS BP Amoco Chemicals Chemicals Research and Engineering, Sunbury-on-Thames Middlesex, TW16 7LL, UK
- SO Organometallics (2000), 19(8), 1470-1476 CODEN: ORGND7; ISSN: 0276-7333
- PB American Chemical Society
- DT Journal
- LA English
- IT 266692-40-4P 266692-41-5P

RL: CAT (Catalyst use); SPN (Synthetic preparation); PREP (Preparation); USES (Uses)

(prepn. and characterization; mechanistic studies of alkene/CO polymn. with palladium complexes promoted by tris(pentafluorophenyl)borane)

RN 266692-40-4 CAPLUS

CN Palladium, [hydroxytris(pentafluorophenyl)borato(1-)-

.kappa.O] (pentafluorophenyl) [1,3-propanediylbis[diphenylphosphine-

.kappa.P]]-, (SP-4-3)- (9CI) (CA INDEX NAME)

PAGE 1-A

PAGE 2-A

RN 266692-41-5 CAPLUS

CN Palladium, [.mu.-hydroxyhexakis(pentafluorophenyl)diborato(1-)-.kappa.O](pentafluorophenyl)[1,3-propanediylbis[diphenylphosphine-.kappa.P]]-, (SP-4-3)- (9CI) (CA INDEX NAME)

PAGE 1-A

PAGE 2-A

PAGE 3-A

AB The reaction of Pd(dppp) (OAc)2 [dppp = 1,3-bis(diphenylphosphino)propane] with B(C6F5)3 in situ gives an efficient catalyst for alkene/CO polymn. in aprotic media. The borane is consumed during the polymn., and its fluoroaryl groups are incorporated into the polymer chain ends. In the absence of the monomers, the catalyst components react to give Pd(II)-pentafluoroaryl complexes formulated as $Pd(dppp)(C6F5)\{[B(C6F5)3]yOH\}\ (y = 1, complex 3a; y = 2, complex 3b).$ Complex 3a can be isolated, albeit as an impure solid, and is itself a catalyst for the reaction. In light of these results, a novel chain initiation process for the polymn. is proposed, involving insertion of monomers into a fluoroarylpalladium complex formed by aryl transfer from the borane to a Pd(II) complex. This facile initiation step, combined with the catalyst stability engendered by the presence of strong Broensted acids, explains the effectiveness of this catalyst system in aprotic media.

RE CNT 42 THERE ARE 42 CITED REFERENCES AVAILABLE FOR THIS RECORD ALL CITATIONS AVAILABLE IN THE RE FORMAT

L4 ANSWER 72 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

AN 2000:164834 CAPLUS

DN 132:308476

TI The Mechanism of Carbon-Carbon Bond Activation in Cationic 6-Alkylcyclohexadienyl Ruthenium Hydride Complexes

AU Older, Christina M.; Stryker, Jeffrey M.

CS Department of Chemistry, University of Alberta, Edmonton, AB, T6G 2G2, Can.

SO Journal of the American Chemical Society (2000), 122(12), 2784-2797 CODEN: JACSAT; ISSN: 0002-7863

PB American Chemical Society

DT Journal

LA English

OS CASREACT 132:308476

IT 264615-83-0P

RL: SPN (Synthetic preparation); PREP (Preparation) (demethylation of hexamethylcyclohexadienyl ruthenium complex with Lewis acids)

RN 264615-83-0 CAPLUS

CN Ruthenium(1+), [(1,2,3,4,5,6-.eta.)-pentamethylbenzene][(1,2,3,4,5-.eta.)-1,2,3,4,5-pentamethyl-2,4-cyclopentadien-1-yl]-, (T-4)-hydroxytris(pentafluorophenyl)borate(1-) (9CI) (CA INDEX NAME)

CM 1

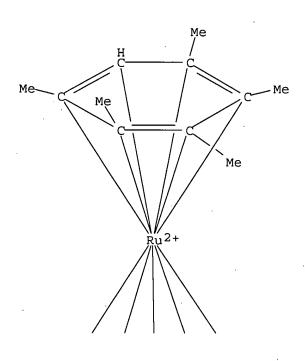
Patel

9/24/2003>

CRN 264615-63-6 CMF C21 H31 Ru

CCI CCS

PAGE 1-A



Me Me C Me

PAGE 2-A

CM 2

CRN 148657-98-1 CMF C18 H B F15 O

CCI CCS

$$F \xrightarrow{F} GH^{-} F \xrightarrow{GH^{-}} F$$

$$F \xrightarrow{F} F$$

$$F \xrightarrow{F} F$$

AB C-C bond activation in cationic 6-endo-methyl-.eta.5-cyclohexadienyl and 6-exo-methyl-.eta.5-cyclohexadienyl Ru hydride complexes was studied. Contrary to expectations, it is the 6-exo-Me complex and not the stereoisomeric 6-endo-Me complex that undergoes selective C-C bond activation under exceptionally mild conditions, quant. converting the 6-exo-Me substituent and the hydride liquid to methane. The mechanism of the activation reaction involves dissocn. of protic acid from the agostic starting complex by reaction with a weak base (typically H2O), followed by protolytic activation of the alkyl group, with backside assistance from the nucleophilic metal center. Under the same conditions, the corresponding 6-endo-Me isomer undergoes selective dehydrogenation rather than demethylation, despite the proximity of the endo-Me substituent to the metal center. For both exo and endo isomers, the cationic Ru hydride intermediates were detd. by spectroscopic anal. to adopt fluxional agostic structures. The agostic complexes are kinetically stable at room temp. under rigorously anhyd. conditions but convert quant. to cationic .eta.6-arene products in the presence of a Bronsted base. The rates of both C-C bond activation and dehydrogenation are dependent on the identity and concn. of the base and suppressed in the presence of excess acid. protolytic mechanism for C-C bond activation is supported by D-labeling studies and by the reactivity of the neutral complexes toward Lewis acids and 1-electron oxidants. This mechanism is relevant to C-C bond activation reactions obsd. in less-substituted 6-exo-methyl-.eta.5cyclohexadienyl complexes and in a steroid-derived 6,6-disubstituted-.eta.5-cyclohexadienyl complex, representative of previously reported cases of dealkylative ligand aromatization. The low kinetic barrier for the protolytic dealkylation mechanism is contrasted to the comparatively high activation barriers reported for C-C bond activation reactions that occur in structurally related systems that cannot access a protolytic pathway. This study provides a consistent basis for rationalizing this potentially important but poorly understood class of metal-mediated reactions.

RE.CNT 130 THERE ARE 130 CITED REFERENCES AVAILABLE FOR THIS RECORD ALL CITATIONS AVAILABLE IN THE RE FORMAT

- L4 ANSWER 73 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN
- AN 2000:159340 CAPLUS
- DN 132:293800
- TI X-ray crystallographic study of three (N.fwdarw.B)-borinates prepared from 8-hydroxyquinoline and 2-hydroxypyridine
- AU Hopfl, Herbert; Barba, Victor; Vargas, Gabriela; Farfan, Norberto; Santillan, Rosa; Castillo, Dolores
- CS Universidad Autonoma del Estado de Morelos, Centro de Investigaciones Quimicas, Mexico, Mex.

Patel 9/24/2003>

Chemistry of Heterocyclic Compounds (New York) (Translation of Khimiya Geterotsiklicheskikh Soedinenii) (2000), Volume Date 1999, 35(8), 912-927 CODEN: CHCCAL; ISSN: 0009-3122

PB Consultants Bureau

DT Journal

LA English

IT 2622-89-1, Diphenylborinic acid

RL: RCT (Reactant); RACT (Reactant or reagent) (condensation reaction with 2-hydroxypyridine)

RN 2622-89-1 CAPLUS

CN Borinic acid, diphenyl- (6CI, 7CI, 8CI, 9CI) (CA INDEX NAME)

GI

8-Hydroxyquinoline and 2-hydroxypyridine were reacted with diphenylborinic acid or 9-BBN to give three heterocyclic products I, II and III. The mol. structure of the resulting heterocycles was studied by x-ray crystallog. (I, space group = P21/c, a = 12.280(1).ANG., b = 17.523(3).ANG., c = 15.158(3).ANG., Z = 8; II, space group = P21/m, a = 8.188(1).ANG., b = 7.005(1).ANG., c = 12.059(1).ANG., Z = 2; III, space group = P21/n, a = 9.505(1).ANG., b = 14.535(1).ANG., c = 22.269(1).ANG., Z = 4). A structural comparison of the so formed five- and six-membered heterocycles with similar complexes obtained from aliph. amino alc. and .alpha.-amino acid derivs. shows significant differences for the N.fwdarw.B, B-O and B-C bond lengths and some of the inner cycle bond angles. Other structural parameters discussed in this respect are the sum of bond lengths at the B

atom, the sum of bond angles in the heterocycle and the tetrahedral character of the B atom. From these parameters a qual. comparison of heterocycle stability is possible.

RE.CNT 58 THERE ARE 58 CITED REFERENCES AVAILABLE FOR THIS RECORD ALL CITATIONS AVAILABLE IN THE RE FORMAT

L4 ANSWER 74 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

AN 2000:84010 CAPLUS

DN 132:237402

TI Silica-Grafted Borato Cocatalysts for Olefin Polymerization Modeled by Silsesquioxane-Borato Complexes

AU Duchateau, Robbert; Van Santen, Rutger A.; Yap, G. P. A.

CS Dutch Polymer Institute/Schuit Institute of Catalysis, Eindhoven University of Technology, Eindhoven, 5600 MB, Neth.

SO Organometallics (2000), 19(5), 809-816 CODEN: ORGND7; ISSN: 0276-7333

PB American Chemical Society

DT Journal

LA English

IT 147892-18-0P 262300-74-3P

RL: SPN (Synthetic preparation); PREP (Preparation) (silica-grafted borato cocatalysts for olefin polymn. modeled by silsesquioxane-borato complexes)

RN 147892-18-0 CAPLUS

CN Borate(1-), hydroxytris(pentafluorophenyl)-, (T-4)-, hydrogen, compd. with N,N-diethylethanamine (1:1) (9CI) (CA INDEX NAME)

CM 1

CRN 147892-17-9 CMF C18 H B F15 O . H CCI CCS

● H+

CM 2

CRN 121-44-8 CMF C6 H15 N

RN 262300-74-3 CAPLUS

CN Borate(1-), hydroxytris(pentafluorophenyl)-, (T-4)-, hydrogen, compd. with N,N-dimethylbenzenamine (1:1) (9CI) (CA INDEX NAME)

CM 1

CRN 147892-17-9 CMF C18 H B F15 O . H CCI CCS

$$F \xrightarrow{F} F \xrightarrow{OH^{-}} F \xrightarrow{F} F$$

$$F \xrightarrow{F} F \xrightarrow{F} F$$

● H+

CM 2

CRN 121-69-7 CMF C8 H11 N

The syntheses and reactivity studies of silsesquioxane-borato complexes are described. Treatment of B(C6F5)3 with (c-C5H9)7Si8O12(OH) and (c-C5H9)7Si7O9(OH)3 in the presence of a Bronsted base yields the silsesquioxane-borates X+{[(c-C5H9)7Si8O13]B(C6F5)3}- (la, X+ = PhN(H)Me2+; lb, X+ = Et3NH+) and X+{[(c-C5H9)7Si7(OH)2O10]B(C6F5)3}- (lb, X+ = PhN(H)Me2+; 2b, X+ = Et3NH+), resp. When the more nucleophilic base pyridine is used, (C6F5)3B.cntdot.NC5H5 (3) is formed instead, demonstrating the competition between B(C6F5)3 and H+ to react with the amine. The dimethylaniline in la and 2a is readily exchanged by NEt3 to form 1b and 2b. With the nucleophilic Lewis base NC5H5, the B-O bond in la and 2a is split, yielding (C6F5)3B.cntdot.NC5H5 (3) and the free

silsesquioxanes. Complexes 1 and 2 rapidly undergo hydrolysis under formation of the hydroxyl complexes $X+\{(C6F5)3BOH\}-(4a, X+=PhN(H)Me2+;4b, X+=Et3NH+)$. Likewise, alcoholysis of 1a and 2a with i-PrOH yields the alkoxide $\{PhN(H)Me2\}+\{i-PrOB(C6F5)3\}-(5)$. The B-O bond is only moderately stable toward early-transition-metal alkyls. Nevertheless, Cp2Zr(CH2Ph)2+1a and Zr(CH2Ph)4+2a form single-site ethylene polymn. catalysts. Detailed reactivity studies demonstrated that both B-O and B-C bond splitting plays a crucial role, as not 1a and 2a, but their decompn. product B(C6F5)3 is the actual cocatalyst. The solid-state structures of 1a and 4b were detd. by single-crystal X-ray anal.

RE.CNT 79 THERE ARE 79 CITED REFERENCES AVAILABLE FOR THIS RECORD ALL CITATIONS AVAILABLE IN THE RE FORMAT

L4 ANSWER 75 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

AN 1999:816392 CAPLUS

DN 132:151856

TI Deprotonation from an N-Methyl Group in 2-[1-(Dimethylamino)-1-methylethyl]phenylborane Derivatives

AU Asakura, Mitsuhiro; Oki, Michinori; Toyota, Shinji

CS Department of Chemistry Faculty of Science, Okayama University of Science, Ridaicho Okayama, 700-0005, Japan

SO Organometallics (2000), 19(2), 206-208 CODEN: ORGND7; ISSN: 0276-7333

PB American Chemical Society

DT Journal

LA English

IT 258280-96-5P

RL: PRP (Properties); SPN (Synthetic preparation); PREP (Preparation) (crystal structure)

RN 258280-96-5 CAPLUS

CN Boron, aquatris[2-[(dimethylamino)methyl]phenyl]-, (T-4)- (9CI) (CA INDEX NAME)

AB Reaction of 2-[1-(dimethylamino)-1-methylethyl]phenyllithium (Ar*Li) with a trialkyl borate, B(OR)3 (e.g., R = iPr), in the 3:1 ratio gave 1-Ar*-3,4,4-trimethyl-1,2,3,4-tetrahydro-3,1-benzazaborin as a major product together with the corresponding protonated compd. and the boronic acid. The structure of the heterocyclic compd. was detd. by x-ray anal. and NMR spectroscopy. This compd. is formed via the deprotonation from one of the N-Me groups in Ar*2B(OR) by the remaining Ar*Li followed by the facile intramol. cyclization between the B and C atoms.

RE.CNT 19 THERE ARE 19 CITED REFERENCES AVAILABLE FOR THIS RECORD

ALL CITATIONS AVAILABLE IN THE RE FORMAT

- L4 ANSWER 76 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN
- AN 1999:789485 CAPLUS
- DN 132:180691
- TI Oxidation of [M(.eta.-C5H5)2], M = Cr, Fe or Co, by the new Bronsted acid H2O.B(C6F5)3 yielding the salts [M(.eta.-C5H5)2]+A-, where A- = [(C6F5)3B(.mu.-OH)B(C6F5)3]- or [(C6F5)3BOH.cntdot..cntdot..cntdot.H2OB(C6F5)3]-
- AU Doerrer, Linda H.; Green, Malcolm L. H.
- CS Inorganic Chemistry Laboratory, Oxford, OX1 3QR, UK
- SO Journal of the Chemical Society, Dalton Transactions: Inorganic Chemistry (1999), (24), 4325-4329
 CODEN: JCDTBI; ISSN: 0300-9246
- PB Royal Society of Chemistry
- DT Journal
- LA English
- RN 155962-45-1 CAPLUS
- CN Boron, aquatris(pentafluorophenyl)-, (T-4)- (9CI) (CA INDEX NAME)

- IT 259684-54-3P 259684-57-6P 259684-62-3P
 - RL: PRP (Properties); SPN (Synthetic preparation); PREP (Preparation) (prepn. and crystal structure of)
- RN 259684-54-3 CAPLUS
- CN Chromocenium, (T-4)-hydroxytris(pentafluorophenyl)borate(1-), compd. with (T-4)-aquatris(pentafluorophenyl)boron and dichloromethane (1:1:1) (9CI) (CA INDEX NAME)
 - CM 1
 - CRN 155962-45-1
 - CMF C18 H2 B F15 O
 - CCI CCS

CM 2

CRN 75-09-2 CMF C H2 Cl2

$Cl-CH_2-Cl$

CM 3

CRN 259684-53-2 CMF C18 H B F15 O . C10 H10 Cr

CM 4

CRN 148657-98-1 CMF C18 H B F15 O CCI CCS

$$F \longrightarrow C \xrightarrow{B} \xrightarrow{S+} C \xrightarrow{F} F$$

$$F \longrightarrow F$$

$$F \longrightarrow F$$

$$F \longrightarrow F$$

$$F \longrightarrow F$$

· CM 5

CRN 12793-15-6 CMF C10 H10 Cr CCI CCS

RN 259684-57-6 CAPLUS

CN Ferrocenium, (T-4)-hydroxytris(pentafluorophenyl)borate(1-), compd. with (T-4)-aquatris(pentafluorophenyl)boron and dichloromethane (2:2:1) (9CI) (CA INDEX NAME)

CM 1

CRN 155962-45-1 CMF C18 H2 B F15 O

CCI CCS

CM 2

CRN 75-09-2 CMF C H2 Cl2

 $C1 - CH_2 - C1$

CM 3

CRN 259684-56-5

CMF C18 H B F15 O . C10 H10 Fe

CM 4

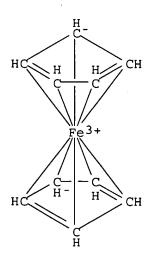
CRN 148657-98-1 CMF C18 H B F15 O

CCI CCS

CM 5

CRN 12125-80-3 CMF C10 H10 Fe

CCI CCS



RN 259684-62-3 CAPLUS

CN Cobaltocenium, (T-4)-hydroxytris(pentafluorophenyl)borate(1-), compd. with (T-4)-aquatris(pentafluorophenyl)boron and dichloromethane (1:1:1) (9CI) (CA INDEX NAME)

CM 1

CRN 155962-45-1

CMF C18 H2 B F15 O CCI CCS

$$F \xrightarrow{F} C^{-} \xrightarrow{OH_2} F \xrightarrow{F} F$$

$$F \xrightarrow{F} F \xrightarrow{F} F$$

CM 2

CRN 75-09-2 CMF C H2 Cl2

 ${\tt Cl-CH_2-Cl}$

CM 3

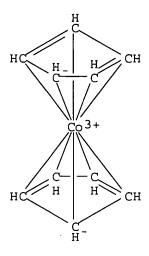
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CM 4

CRN 148657-98-1 CMF C18 H B F15 O CCI CCS

CM 5

CRN 12241-42-8 CMF C10 H10 Co CCI CCS



$$F \longrightarrow F \longrightarrow F \longrightarrow F$$

$$F \longrightarrow F \longrightarrow F$$

$$F \longrightarrow F$$

$$F \longrightarrow F$$

$$F \longrightarrow F$$

RN 259684-66-7 CAPLUS
CN Chromocenium, .mu.-hydroxyhexakis(pentafluorophenyl)diborate(1-) (9CI) (CA INDEX NAME)

CM 1

CRN 219697-03-7

CMF C36 H B2 F30 O

CCI CCS

PAGE 1-A

PAGE 2-A

PAGE 3-A

CM 2

CRN 12793-15-6 CMF C10 H10 Cr CCI CCS

RN 259684-69-0 CAPLUS

CN Cobaltocenium, .mu.-hydroxyhexakis(pentafluorophenyl)diborate(1-) (9CI) (CA INDEX NAME)

CM 1

CRN 219697-03-7

CMF C36 H B2 F30 O

CCI CCS

PAGE 1-A

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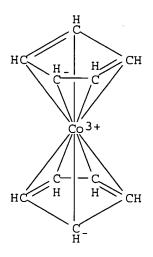
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PAGE 2-A

PAGE 3-A

CM 2

CRN 12241-42-8 CMF C10 H10 Co CCI CCS



The Bronsted acids H2O.B(C6F5)3 and D2O.B(C6F5)3 were synthesized.

Reaction of neutral divalent metallocenes [M(.eta.-C5H5)2], M = Cr, Fe or Co, with two equiv. of H2O.B(C6F5)3 (2a) resulted in metallocene oxidn. and formation of salts contg. [M(.eta.-C5H5)2] + cations together with the hydroxoborate anion [HOB(C6F5)3] - which is H bonded to the 2nd acid equiv., [M(.eta.-C5H5)2] [(F5C6)3BOH.cntdot..cntdot..cntdot.H2OB(C6F5)3], M = Cr (3a), Fe (4a) or Co (5a). Treatment of one equiv. of 2a and one equiv. of B(C6F5)3 with [M(.eta.-C5H5)2] yielded salts contg. the same metallocene cations but now with .mu.-OH bridged anions, as in [M(.eta.-C5H5)2] [(F5C6)3B(.mu.-OH)B(C6F5)3], where M = Cr or Co. All products were characterized by NMR spectroscopy, elemental anal., and the single-crystal structures of 2a, 3a, 4a, and 5a were detd.

RE.CNT 36 THERE ARE 36 CITED REFERENCES AVAILABLE FOR THIS RECORD

Patel

9/24/2003>

ALL CITATIONS AVAILABLE IN THE RE FORMAT

- L4 ANSWER 77 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN
- AN 1999:779977 CAPLUS
- DN 132:137431
- TI Boron and transition metal compounds derived from 2-uroylbenzimidazole
- AU Fialon, Marie-Pierre; Garcia-Baez, Efren; Andrade-Lopez, Noemi; Osorio-Monreal, Guadalupe; Canseco-Melchor, Graciela; Velazquez-Montes, Imelda; Barba-Behrens, Norah; Contreras, Rosalinda
- CS Departamento de Quimica, Centro de Investigacion y de Estudios Avanzados del IPN, Mexico, 07000, Mex.
- SO Heteroatom Chemistry (1999), 10(7), 577-584 CODEN: HETCE8; ISSN: 1042-7163
- PB John Wiley & Sons, Inc.
- DT Journal
- LA English
- OS CASREACT 132:137431
- IT 2622-89-1, Diphenylborinic acid
 RL: RCT (Reactant); RACT (Reactant or reagent)
 (cyclization with uroylbenzimidazole)
- RN 2622-89-1 CAPLUS
- CN Borinic acid, diphenyl- (6CI, 7CI, 8CI, 9CI) (CA INDEX NAME)

GI

- The coordination sites of 2-uroylbenzimidazole (1; uroyl = NHC(O)NH2) toward diphenylborinic acid, as well as Zn, Cd, Cu, Ni and Co chlorides, bromides, and nitrates were studied. The ligand is bonded in monodentate mode to ZnCl2, ZnBr2, CdCl2, CdBr2, and Cd(NO3)2, and in bidentate mode to all others. With diphenylborinic acid, two heterocycles are formed; in one, the B is bonded to imidazole and the terminal NH2 group, and in the other the B is bonded to imidazole and to the O atom; only I was isolated. B, Zn, and Cd derivs. were studied by NMR spectroscopy. The x-ray diffraction structures of 2-uroylbenzimidazole and L2CoCl2 (L = Me 2-benzimidazolylcarbamate) are reported.
- RE.CNT 9 THERE ARE 9 CITED REFERENCES AVAILABLE FOR THIS RECORD ALL CITATIONS AVAILABLE IN THE RE FORMAT
- L4 ANSWER 78 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN
- AN 1999:764365 CAPLUS
- DN 132:12606

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TΙ
    Catalyst system and its use for polymerizing propylene
IN
    Bohnen, Hans; Goeres, Markus; Fritze, Cornelia
    Aventis Research und Technologies G.m.b.H. und Co. K.-G., Germany
PA
SO
    Ger. Offen., 16 pp.
    CODEN: GWXXBX
DT
    Patent
    German
LA
FAN.CNT 2
    PATENT NO.
                    KIND DATE
                                         APPLICATION NO. DATE
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PΙ
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                                         DE 1998-19823171A 19980523
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    JP 2002516358
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            PT, SE
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                                         WO 1999-EP3416 W 19990518
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                                         WO 1999-EP3416 W 19990518
OS
    MARPAT 132:12606
IT
    2118-02-7
    RL: RCT (Reactant); RACT (Reactant or reagent)
       (reactant; in prepn. of metallocene catalyst systems for polymn. of
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propylene)

RN 2118-02-7 CAPLUS

CN Borinic acid, bis(pentafluorophenyl) - (7CI, 8CI, 9CI) (CA INDEX NAME)

AB Cost effect highly active aluminoxane-free metallocene catalyst systems for the polymn. of propylene contain .qtoreq.1 metallocene as a rac-meso isomeric mixt., .gtoreq.1 organoboraluminum compd., .gtoreq.1 inert org. or inorg. carrier, .gtoreq.1 Lewis base, and, optionally, .gtoreq.1 addnl. organometal compd. Polymn. proceeds without formation of deposits on the reactor walls and polypropylene with a high m.p. and tacticity is produced. Hydrogen can be used in the polymn. as a mol. wt. regulator or to increase the activity of the catalyst systems. Thus, treatment of trimethylaluminum with pentafluoroboronic acid in toluene gave a clear colorless soln. of bis(pentafluorophenylboroxy)methylalane which was added dropwise to a toluene suspension of silica which had been pretreated with N, N-dimethylaniline. The solvent was evapd. to give a support material which was mixed with a toluene soln. of dimethylsilandiylbis(2-n-propyl-4-(4'-tert-butylphenyl)indenyl)zirconium dichloride (rac/meso ratio 1:1.5 .mu.mol) and trimethylaluminum. Evapn. of the solvent gave a free-flowing rose-colored powder which was used with triisobutylaluminum to polymerize propylene. No deposits were obsd. on the inner walls of the polymn. reactor. The catalyst activity was 161 kg polypropylene/g metallocene/h.

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L4 ANSWER 79 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN
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AN 1999:764364 CAPLUS

DN 132:12587

TI Use of metallocene-containing catalyst system for olefin polymerization

IN Bohnen, Hans; Goeres, Markus; Fritze, Cornelia

PA Aventis Research und Technologies G.m.b.H. und Co. K.-G., Germany

SO Ger. Offen., 14 pp.

CODEN: GWXXBX

DT Patent

LA German

FAN. CNT 2

FAN.	CNT 2		
	PATENT NO. KIND DAT	E APPLICATION NO.	DATE
ΡI	DE 19823171 A1 199	91125 DE 1998-19823171	19980523
	WO 9961487 A1 199	91202 WO 1999-EP3416	19990518
	W: BR, CN, JP, KR, US	}	•
	RW: AT, BE, CH, CY, DE	C, DK, ES, FI, FR, GB, GR, IE	, IT, LU, MC, NL,
	PT, SE		
		DE 1998-198231712	A 19980523
		DE 1998-19823172	A 19980523
	EP 1082353 A1 200	10314 EP 1999-953341	19990518
	R: DE, ES, FR, GB, IT	NL ·	
		DE 1998-198231712	A 19980523
		DE 1998-19823172	A 19980523

	JP 2002516358	T2 20020)604 JF DE	WO 1999-EP3416 W 19990518 JP 2000-550890 19990518 DE 1998-19823171A 19980523 DE 1998-19823172A 19980523		
	US 6576723	B1 20030	WC D610 US DE DE	1999-EP3416 W 2000-700425 1998-198231712 1998-198231722 1999-EP3416 W	19990518 20001115 A 19980523 A 19980523	
PATE	NT FAMILY INFORMATI	ON:	***	, 1999 PL9410 W	1,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,	
FAN		IND DATE	AF	PLICATION NO.	DATE	
ΡI		71 1000	1125 75	1998-19823172	10000522	
PI	WO 9961487					
	W: BR, CN, JP			2777 213110	1,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,	
			DK, ES, FI,	FR, GB, GR, IE,	IT, LU, MC, NL,	
				1998-19823171		
				1998-19823172		
•	EP 1082353 R: DE, ES, FR			1999-953341	19990518	
			DE	: 1998-19823171 : 1998-19823172	A 19980523	
	JP 2002516358	TO 2002		1999-EP3416 W 2000-550890		
	UP 2002516356	12 20020		: 1998-19823171 <i>i</i>		
				: 1998-19823172 <i>I</i>		
	-			1999-EP3416 W		
	US 6576723	B1 20030		2000-700425		
				1998-19823171		
	·		DE	1998-19823172	A 19980523	
			WC	1999-EP3416 W	19990518	
OS IT	OS MARPAT 132:12587					
	metallocene-con	itg. cataly	yst systems f	or polymn. of o	olefins)	
RN CN	2118-02-7 CAPLUS Borinic acid, bis(pentafluo	rophenyl)- (7	CI, 8CI, 9CI)	(CA INDEX NAME)	

AB Cost effective, highly active catalyst systems for the polymn. of olefins contain .gtoreq.1 metallocene as a rac-meso isomeric mixt., .gtoreq.1 organoboraluminum compd., .gtoreq.1 org. or inorg. inert support, .gtoreq.1 Lewis base, and, optionally, .gtoreq.1 addnl. organometal compd. The catalyst systems can be used to polymn. olefins without deposit formation on the walls of the polymn. reactor and yield polymers with a

homogeneous granular morphol. The use of hydrogen in polymn. significantly increases the activity of the catalyst systems. Thus, treatment of trimethylaluminum with pentafluoroboronic acid in toluene gave a bis (pentyfluorophenylboroxy) methylalane soln. which was added dropwise to a toluene suspension of silica which had been pretreated with N,N-dimethylaniline. The solvent was evapd. to give a support material which was mixed with toluene solns. of dimethylsilandiylbis(2-n-propyl-4-(4'-tert-butylphenyl)-indenyl)zirconium dichloride (rac/meso ratio 1:1.5 .mu.mol) and trimethylaluminum. Evapn. of the solvent gave a rose-colored free-flowing powder.

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L4 ANSWER 80 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN
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AN 1999:764363 CAPLUS

DN 132:12586

TI Catalyst system for olefin polymerization

IN Bohnen, Hans; Goeres, Markus; Fritze, Cornelia

PA Aventis Research und Technologies G.m.b.H. und Co. K.-G., Germany

SO Ger. Offen., 18 pp.

CODEN: GWXXBX

DT Patent

LA German

FAN.CNT 1

	PATENT NO.	. KIND DATE	APPLICATION NO. DATE
			
ΡI	DE 19823168	A1 19991125	DE 1998-19823168 19980523
	WO 9961488	A1 19991202	WO 1999-EP3415 19990518
	W: BR, CI	N, JP, KR, US	•
	RW: AT, B	E, CH, CY, DE, DK, 1	ES, FI, FR, GB, GR, IE, IT, LU, MC, NL,
	PT, S	E	
			DE 1998-19823168A 19980523
	BR 9910675	A 20010130	BR 1999-10675 19990518
			DE 1998-19823168A 19980523
			WO 1999-EP3415 W 19990518
•	EP 1086146	A1 20010328	EP 1999-925004 19990518
	R: BE, D	E, ES, FR, GB, IT, 1	NL, FI
			DE 1998-19823168A 19980523
			WO 1999-EP3415 W 19990518
	JP 2002516359	T2 20020604	JP 2000-550891 19990518
		•	DE 1998-19823168A 19980523

OS MARPAT 132:12586

IT 2118-02-7, Bis(pentafluorophenyl)borinic acid 2622-89-1

RL: RCT (Reactant); RACT (Reactant or reagent)

(reactant; in prepn. of organoboraluminum compds. for use as catalyst components for polymn. of olefins)

WO 1999-EP3415 W 19990518

RN 2118-02-7 CAPLUS

CN Borinic acid, bis(pentafluorophenyl) - (7CI, 8CI, 9CI) (CA INDEX NAME)

RN 2622-89-1 CAPLUS

CN Borinic acid, diphenyl- (6CI, 7CI, 8CI, 9CI) (CA INDEX NAME)

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Ph
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|
Ph— B— OH
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AB Catalyst systems contg. .gtoreq.1 metallocene, .gtoreq.1 Lewis base, a porous inorg. or org. support, .gtoreq.1 organoboraluminum compd., and, optionally, an organometallic compd. can be used for the polymn. of olefins without the use of the excess aluminoxanes usually needed as cocatalysts and still attain a high catalyst activity and good polymer morphol. A significant increase of the catalyst activity is produced by the addn. of hydrogen during polymn. Thus, treatment of trimethylaluminum with pentafluoroboronic acid in toluene yielded a clear bright yellow soln. of bis(dimethylalumoxy)pentafluorophenylborane which was then dropped into a toluene suspension of SiO2 which had been pretreated with N,N-dimethylaniline. The blue colored support material obtained by drying the above suspension was then mixed with toluene solns. of dimethylsilandiylbis(2-methyl-4-phenylindenyl)zirconium di-Me and trimethylaluminum and the solvent was evapd. to give a rose-colored free-flowing powder. Propylene was polymd. using this powder and triisobutylaluminum as catalysts. No deposits occurred on the inner wall of the polymn. reactor. The catalyst activity was 26 kg polypropylene/g metallocene/h.

L4 ANSWER 81 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

AN 1999:718943 CAPLUS

DN 131:339340

TI Method of making an improved catalyst containing an acid-treated zeolite, a boron component, and a zinc component, a product from such method, and the use thereof in the conversion of hydrocarbons

IN Drake, Charles A.

PA Phillips Petroleum Co., USA

SO U.S., 8 pp. CODEN: USXXAM

DT Patent

LA English

FAN.CNT 1

1111.	CIVI				
	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
					-
ΡI	US 5981417	Α	19991109	US 1998-40704	19980318
	US 6107534	Α	20000822	US 1999-384365	19990825
				IIS 1998-40704	A319980318

IT 12113-07-4

RL: CAT (Catalyst use); RCT (Reactant); RACT (Reactant or reagent); USES (Uses)

(zeolite catalysts contg.; method of making improved catalysts contg. acid-treated zeolites, boron and zinc components, products from such method, and use in conversion of hydrocarbons)

RN 12113-07-4 CAPLUS

CN Borate(1-), hydroxytriphenyl-, sodium, (T-4)- (9CI) (CA INDEX NAME)

Na+

AB The invention relates to an improved zeolite catalyst contg. an acid-treated zeolite, a boron component and a zinc component manufd. by a novel method having certain process steps necessary for providing the improved zeolite catalyst. The process steps include a first steam treatment of an acid-treated zeolite, followed by incorporation of such zeolite with a boron component and a zinc component, followed by a second steam treatment. Processes are also disclosed for using the improved zeolite catalyst in the conversion of hydrocarbons, preferably nonarom. hydrocarbons, to lower olefins (such as ethylene and propylene) and arom. hydrocarbons (such as benzene, toluene, and xylene).

RE.CNT 4 THERE ARE 4 CITED REFERENCES AVAILABLE FOR THIS RECORD ALL CITATIONS AVAILABLE IN THE RE FORMAT

L4 ANSWER 82 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

AN 1999:596212 CAPLUS

DN 131:337160

TI Electrophilic Binuclear Methylplatinum(II) Complexes

AU Baar, Cliff R.; Jennings, Michael C.; Puddephatt, Richard J.; Muir, Kenneth W.

CS Department of Chemistry, University of Western Ontario, London, ON, N6A 5B7, Can.

SO Organometallics (1999), 18(21), 4373-4379 CODEN: ORGND7; ISSN: 0276-7333

PB American Chemical Society

DT Journal

LA English

OS CASREACT 131:337160

IT 249643-57-0P

RL: SPN (Synthetic preparation); PREP (Preparation)
 (bo sd)

RN 249643-57-0 CAPLUS

CN Platinum(2+), bis(acetonitrile)[rel-(1R,2R)-N,N'-bis[(2-pyridinyl-.kappa.N)methylene]-1,2-cyclohexanediamine-.kappa.N,.kappa.N']dimethyldi-, stereoisomer, bis[(T-4)-hydroxytris(pentafluorophenyl)borate(1-)] (9CI) (CA INDEX NAME)

CM 1

CRN 249643-56-9 CMF C24 H32 N6 Pt2 CCI CCS

CM 2

CRN 148657-98-1 CMF C18 H B F15 O CCI CCS

IT 250129-66-9P

RN 250129-66-9 CAPLUS

CN Platinum(2+), bis(acetonitrile)[.mu.-[rel-(1R,2S)-N,N'-bis[(2-pyridinyl-.kappa.N)methylene]-1,2-cyclohexanediamine-.kappa.N:.kappa.N']]di-, stereoisomer, bis[(T-4)-hydroxytris(pentafluorophenyl)borate(1-)] (9CI) (CA INDEX NAME)

CM 1

CRN 250129-65-8 CMF C24 H32 N6 Pt2

CCI CCS

CM 2

CRN 148657-98-1 CMF C18 H B F15 O CCI CCS

The bis(bidentate) ligands trans- and cis-1,2-C6H10(N:CH-2-C5H4N)2 (1 and 2) yield the diplatinum(II) complexes trans- and cis-1,2-[C6H10 $\{N:CH-2-C5H4N(PtMe2)\}2\}$ (3 and 4, resp.). Reaction of 3 and 4 with [H]+[HOB(C6F5)3]- in MeCN or with HBF4 in the presence of excess CO gave the corresponding electrophilic binuclear complexes trans- and cis-1,2-[C6H10 $\{N:CH-2-C5H4N(PtMeL)\}2\}$ [X]2 (5, trans, L = MeCN, X = [HOB(C6F5)3]; 6, cis, L = MeCN, X = [HOB(C6F5)3]; 7, trans, L = CO, X = BF4; 8, cis, L = CO, X = BF4). The electrophilic complexes 5-8 are formed by selective Me group protonolysis, and the stereochemistries were confirmed for complexes 7 and 8 by x-ray structure detns.

RE.CNT 49 THERE ARE 49 CITED REFERENCES AVAILABLE FOR THIS RECORD ALL CITATIONS AVAILABLE IN THE RE FORMAT

L4 ANSWER 83 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

AN 1999:530955 CAPLUS

DN 131:170502

TI Preparation of aldehydes or ketones with aryl boron compound catalysts

IN Yamamoto, Takashi; Ishihara, Kazuaki; Kurihara, Hideki

PA Nagoya University, Japan

SO Jpn. Kokai Tokkyo Koho, 9 pp.

CODEN: JKXXAF

DT Patent

LA Japanese

FAN.	CNT	

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI.	JP 11228479	A2	19990824	JP 1998-30940	19980213
	JP 2884081	B2	19990419	31 2773 33713	
	JP 2884081	B1	19990419		

JP 1998-30940 19980213

OS CASREACT 131:170502; MARPAT 131:170502

IT 2118-02-7

RL: CAT (Catalyst use); USES (Uses)

(prepn. of aldehydes or ketones by oxidn. of alcs. in the presence of aryl boron compds. catalysts)

RN 2118-02-7 CAPLUS

CN Borinic acid, bis(pentafluorophenyl) - (7CI, 8CI, 9CI) (CA INDEX NAME)

GΙ

$$R^{5}$$
 $C-R^{6}$
 R^{7}
 R^{7}

AB Perillaldehyde, R2CR1:CHR3CHO [R1, R2 = H, (halo-substituted) C1-10 branched or linear (un)satd. aliph. hydrocarbyl; R3 = C1-4 alkyl, H], arom. ketones I (X = H, halo, C1-4 alkyl; R4 = H; R5 = H, halo, C1-4 alkyl; R6, R7 = H, R4R5 = benzene ring; R6R7 = 5-membered ring), myrtenal, or (1S)-(-)-verbenone is prepd. by oxidn. of perillyl alc.,

R2CR1:CHR3CHR4OH (R1-R3 = same as above), arom. alcs. II (R4-R7, X = same as I), myrtenol, or (S)-cis-verbenol in the presence of ArnB(OH)3-n (Ar = pentafluorophenyl; n = 2-3). (S)-perillyl alc. was oxidized in the presence of bis(pentafluorophenyl)boric acid, MgSO4, and t-BuCHO in PhMe at room temp. for 3 h to give 99% perillaldehyde.

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ANSWER 84 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN
L4
AN
     1999:511196 CAPLUS
DN
     131:170746
ΤI
     Metallocene catalysts for polymerization of olefins
IN
     Bohnen, Hans; Fritze, Cornelia
PA
     Targor G.m.b.H., Germany
SO
     PCT Int. Appl., 86 pp.
     CODEN: PIXXD2
DT
     Patent
     German
LΑ
FAN.CNT 1
     PATENT NO.
                      KIND
                                            APPLICATION NO.
PΙ
                       A1
                            19990812
                                            WO 1999-EP725
                                                             19990205
         W: BR, CA, CN, GD, IN, JP, KR, NO; RU, US
         RW: AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL,
             PT, SE
                                            DE 1998-19804970A 19980207
     DE. 19804970
                            19990812
                       A1
                                            DE 1998-19804970 19980207
     ZA 9900919
                       Α
                            20000807
                                            ZA 1999-919
                                                             19990205
                                            DE 1998-19804970A 19980207
     EP 1053263
                       A1
                            20001122
                                            EP 1999-907496
                                                             19990205
         R: BE, DE, ES, FR, GB, IT, NL, FI
                                            DE 1998-19804970A 19980207
                                            WO 1999-EP725 W 19990205
     BR 9914227
                                            BR 1999-14227
                       Α
                            20010626
                                                             19990205
                                            DE 1998-19804970A 19980207
                                            WO 1999-EP725 W 19990205
     JP 2002502896
                       T2
                            20020129
                                            JP 2000-530556
                                                             19990205
                                            DE 1998-19804970A 19980207
                                            WO 1999-EP725 W 19990205
     US 6482902
                       В1
                            20021119
                                            US 2000-600313
                                                             20000713
                                            DE 1998-19804970A 19980207
                                            WO 1999-EP725 W 19990205
OS
     MARPAT 131:170746
     2622-89-1P, Diphenylborinic acid
IT
     RL: CAT (Catalyst use); IMF (Industrial manufacture); PREP (Preparation);
     USES (Uses)
        (reaction with trimethylaluminum)
RN
     2622-89-1 CAPLUS
CN
     Borinic acid, diphenyl- (6CI, 7CI, 8CI, 9CI) (CA INDEX NAME)
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Ph
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Ph— B— OH
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CN Borinic acid, bis(pentafluorophenyl) - (7CI, 8CI, 9CI) (CA INDEX NAME)

AB The title catalysts, having high activity and giving polymers with good morphol., contain metallocenes, Lewis bases and org. B-Al compds. of specified structure, supports, and, optionally, organometallic compds. of Group IA, IIA, or IIIA elements. Stirring 1.5 L liq. C3H6 with 3 mL iso-Bu3Al (20% in Varsol) for 15 min, adding 5.8 mg (dimethylsilanediyl)bis(2-methyl-4-phenylindenyl)zirconiumdimethyl, 0.5 g supported bis(dimethylaluminoxy)pentafluorophenylborane, and 20 .mu.mol AlMe3, and stiring for 1 h at 60.degree. gave 151 g powd. polypropylene (26 kg/g metallocene-h).

RE.CNT 5 THERE ARE 5 CITED REFERENCES AVAILABLE FOR THIS RECORD ALL CITATIONS AVAILABLE IN THE RE FORMAT

L4 ANSWER 85 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

AN 1999:459403 CAPLUS

DN 131:214382

- TI The One-Electron Oxidation of an Azazirconacyclobutene in the Presence of B(C6F5)3
- AU Harlan, C. Jeff; Hascall, Tony; Fujita, Etsuko; Norton, Jack R.
- CS Department of Chemistry, Columbia University, New York, NY, 10027, USA
- SO Journal of the American Chemical Society (1999), 121(31), 7274-7275 CODEN: JACSAT; ISSN: 0002-7863
- PB American Chemical Society

DT Journal

LA English

IT 155962-45-1, Aquatris(pentafluorophenyl)boron RL: RCT (Reactant); RACT (Reactant or reagent)

(one-electron oxidn. of azazirconacyclobutene in presence of)

RN 155962-45-1 CAPLUS

CN Boron, aquatris(pentafluorophenyl)-, (T-4)- (9CI) (CA INDEX NAME)

AB Cp2Zr(PhC:CPhNtBu-C,N) (1) undergoes partial (< 1%) oxidn. in the presence of B(C6F5)3. 1 Could be electrochem. oxidized. to 1.bul.+ (E.degree.1/2 = -0.07 vs. SCE in CH2Cl2 (Bu4NPF6, 0.1 N; 50 mV/s)). The electron exchange rate const. for 1 + 1.bul.+ is 1.5 .times. 108 M-1 s-1 at 293 K, implying a hyperfine atBu = 0.071 G and that for C5H5 protons = 0.018 G. In an effort to generate a radical anion from B(C6F5)3, it was treated with cobaltocene to give [Cp2Co][HB(C6F5)3] and CpCo(.eta.5-C5H4B(C6F5)3), the latter of which was characterized by x-ray crystallog. 1.bul.+ Was also formed from 1 and ferricinium, (H2O)B(C6F5)3, [PhNMe2H][B(C6F5)4], or Me aluminoxane.

RE.CNT 29 THERE ARE 29 CITED REFERENCES AVAILABLE FOR THIS RECORD ALL CITATIONS AVAILABLE IN THE RE FORMAT

L4 ANSWER 86 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

AN 1999:434462 CAPLUS

DN 131:184996

TI Dimesitylaminoboranes and unsymmetric triaminoboranes. Stability of aminodioxaboroles and dimesitylboroxyethanol

AU Maarouf, Z. Ben; Chazalette, C.; Riviere-Baudet, M.; Riviere, P.

CS Laboratoire de Chimie Organique et Organometallique, Universite Ibnou Zohr, Agadir, Morocco

SO Main Group Metal Chemistry (1999), 22(6), 405-412 CODEN: MGMCE8; ISSN: 0792-1241

PB Freund Publishing House Ltd.

DT Journal

LA English

IT 20631-84-9P

RN 20631-84-9 CAPLUS

CN Borinic acid, bis(2,4,6-trimethylphenyl) - (9CI) (CA INDEX NAME)

Bulky dimesitylaminoboranes, unsym. triaminoboranes and an amino boron sulfonamide were prepd. either by transmetalation or transamination reactions. From tris(diethylamino) borane, diethylaminodioxaborole was obtained either by protic cleavage by 3,5-di-t-butylcatechol or by addn. reaction of 3,5-di-t-butyl-o-quinone through S.E.T. in the first step of the reaction. From the same tris(diethylamino) borane, 1,2-ethanediol did not lead to the expected diethylaminodioxaborolane but to 2,5,7,10,11,14-hexaoxa-1,6-diborane bicyclo[4.4.4] tetradecane. 2-Dimesitylboroxyethanol, isolated as a white powder, is not thermally stable and leads either to 1,2-bis(dimesitylboroxy) ethane or to 1,3-dioxaborolane with mesitylene elimination. Dimesitylboranes undergo nucleophilic substitution of a mesityl group in the presence of a strong nucleophile.

RE.CNT 15 THERE ARE 15 CITED REFERENCES AVAILABLE FOR THIS RECORD ALL CITATIONS AVAILABLE IN THE RE FORMAT

L4 ANSWER 87 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

- AN 1999:409650 CAPLUS
- DN 131:157779
- TI Copper-catalyzed sodium tetraphenylborate, triphenylborane, diphenylborinic acid and phenylboronic acid decomposition kinetic studies in aqueous alkaline solutions
- AU Crawford, C. L.; Barnes, M. J.; Peterson, R. A.; Wilmarth, W. R.; Hyder, M. L.
- CS Savannah River Site, Westinghouse Savannah River Company, Aiken, SC, USA
- SO Journal of Organometallic Chemistry (1999), 581(1-2), 194-206 CODEN: JORCAI; ISSN: 0022-328X
- PB Elsevier Science S.A.
- DT Journal
- LA English
- IT 2622-89-1, Diphenylborinic acid

RL: PEP (Physical, engineering or chemical process); RCT (Reactant); PROC (Process); RACT (Reactant or reagent)

(copper-catalyzed decompn. kinetics of sodium tetraphenylborate, triphenylborane, diphenylborinic acid, and phenylboronic acid in aq. alk. solns.)

- RN 2622-89-1 CAPLUS
- CN Borinic acid, diphenyl- (6CI, 7CI, 8CI, 9CI) (CA INDEX NAME)

Ph | |-Ph— B— OH

AΒ This work studied the kinetics of copper-catalyzed decompn. of tetraphenylborate, triphenylborane, diphenylborinic acid and phenylboronic acid (NaTPB, 3PB, 2PB and 1PB, resp.) in aq. alk. soln. over the temp. range of 25-70.degree. The statistically designed test matrixes added copper sulfate to max. concns. of 10 mg/l. The relative rates of decompn. increase in the order NaTPB < 1PB .apprx. 3PB < 2PB. Dependence of decompn. on the amt. of added copper increases in the order 3PB .apprx. 2PB < 1PB .apprx. NaTPB. Activation energies ranged from 82-143 kJ mol-1 over the temp. range studied. Final decompn. products involved benzene and phenol predominately. All 3PB, 2PB and 1PB intermediate phenylborate species proved relatively stable (< 8% decompn. over ca. 500 h) towards thermal hydrolysis in 1.5 M NaOH when contained in carbon-steel vessels sealed under air at ambient temp. (23-25.degree.) with no added copper. Measurable (> 10-7 M h-1) thermal hydrolysis of the phenylborate species occurs at 55-70.degree. in alk. (0.6-2.3 M OH-, 2-4.7 M Na+) soln. with no added copper. The expts. suggest an important role for oxygen in copper-catalyzed phenylborate decompn. NaTPB decomps. promptly under anoxic conditions while 3PB, 2PB and 1PB decomp. faster in aerobic solns. Benzene and phenol form as the predominant end-products from alk. copper catalysis in static systems sealed under air. Both 2PB and 1PB decomp. with near equal rates and quant. produce phenol under flowing air-purge conditions at 25-60.degree... Mechanisms for copper-catalyzed phenylborate decompn. likely involve a redox process giving loss of a Ph group from the phenylborate with redn. of cupric ion, or dephenylation by reduced cuprous ion involving a phenylated copper intermediate.

RE.CNT 43 THERE ARE 43 CITED REFERENCES AVAILABLE FOR THIS RECORD ALL CITATIONS AVAILABLE IN THE RE FORMAT

L4 ANSWER 88 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN AN 1999:384961 CAPLUS

DN 131:210950

TI Protonation of the Hydroxide Ligand in a Synthetic Analogue of Carbonic Anhydrase, [TpBut,Me] ZnOH: Inhibition of Reactivity Towards CO2

AU Bergquist, Catherine; Parkin, Gerard

CS Department of Chemistry, Columbia University, New York, NY, 10027, USA

SO Journal of the American Chemical Society (1999), 121(26), 6322-6323 CODEN: JACSAT; ISSN: 0002-7863

PB American Chemical Society

DT Journal

LA English

IT 243121-77-9P

RL: BSU (Biological study, unclassified); SPN (Synthetic preparation); BIOL (Biological study); PREP (Preparation)

(protonation of the hydroxide ligand in a synthetic analog of carbonic anhydrase causes inhibition of reactivity towards carbon dioxide)

RN 243121-77-9 CAPLUS

CN Zinc(1+), aqua[tris[3-(1,1-dimethylethyl)-5-methyl-1H-pyrazolato-.kappa.N1]hydroborato(1-)-.kappa.N2,.kappa.N2',.kappa.N2'']-, (T-4)-, (T-4)-hydroxytris(pentafluorophenyl)borate(1-) (9CI) (CA INDEX NAME)

CM 1

CRN 243121-76-8

CMF C24 H42 B N6 O Zn

cci ccs

Me N Bu-t
$$2n^{2+}$$
 OH_2 Me N Bu -t Bu -t

CM 2

CRN 148657-98-1

CMF C18 H B F15 O

CCI CCS

IT 147892-17-9

RL: RCT (Reactant); RACT (Reactant or reagent)
(protonation of the hydroxide ligand in a synthetic analog of carbonic anhydrase causes inhibition of reactivity towards carbon dioxide)

$$F \xrightarrow{F} F \xrightarrow{OH^{-}} F \xrightarrow{F} F$$

$$F \xrightarrow{F} F \xrightarrow{F} F$$

● H+

An essential step in the mechanisms of action of a large variety of zinc enzymes, such as carbonic anhydrase, involves reversible proton transfer which serves to interconvert the aqua and hydroxide forms of the active sites, [LZn-OH2]2+ and [LZn-OH]+. Surprisingly, however, examples of this transformation for which both partners have been isolated and structurally characterized are unknown. In this paper, we report the synthesis and structural characterization of a monomeric zinc aqua complex, which is obtained by protonation of the hydroxide form of a synthetic analog of carbonic anhydrase, and also demonstrate that protonation inhibits reactivity towards CO2.

RE.CNT 53 THERE ARE 53 CITED REFERENCES AVAILABLE FOR THIS RECORD ALL CITATIONS AVAILABLE IN THE RE FORMAT

L4 ANSWER 89 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

AN 1999:261740 CAPLUS

DN 131:5286

TI NMR structural analysis of boron derivatives of 2-guanidinobenzimidazole

- AU Andrade-Lopez, N.; Tlahuext, H.; Contreras, R.
- CS Departamento de Quimica, Centro de Investigación y de Estudios Avanzados del Instituto Politecnico Nacional, Mexico, 07000, Mex.
- SO Congreso Iberoamericano de Quimica Inorganica, 6th, Puebla, Mex., Apr. 20-25, 1997 (1997), 274-277 Publisher: Asociacion Mexicana de Quimica Inorganica, Guanajuato, Mex. CODEN: 67NIAA
- DT Conference
- LA Spanish
- IT 2622-89-1, Diphenylborinic acid

RL: RCT (Reactant); RACT (Reactant or reagent)

(NMR structural anal. of boron derivs. of guanidinobenzimidazole)

- RN 2622-89-1 CAPLUS
- CN Borinic acid, diphenyl- (6CI, 7CI, 8CI, 9CI) (CA INDEX NAME)

- AB A symposium article with 2 refs. Mol. structures of the title compds., formed by reaction of 2-guanidinobenzimidazole with RR1BOH (R = R1 = Ph, F, OH; R = Ph, R1 = OH, OMe), were examd. in soln. by NMR and by reactions with acids and water. All 5 products exist as equil. mixts. of tautomers in soln., with one predominating. The structure of the compd. prepd. from Ph2BOH was detd. by x-ray crystallog.
- RE.CNT 2 THERE ARE 2 CITED REFERENCES AVAILABLE FOR THIS RECORD ALL CITATIONS AVAILABLE IN THE RE FORMAT
- L4 ANSWER 90 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN
- AN 1999:261730 CAPLUS
- DN 131:5285
- TI Bora heterocycles derived from 2-guanidinobenzimidazole
- AU Cartas-Rosado, A. R.; Andrade-Lopez, N.; Contreras, R.
- CS Departamento de Quimica, Centro de Investigacion y de Estudios Avanzados del Instituto Politecnico Nacional, Mexico, 07000, Mex.
- SO Congreso Iberoamericano de Quimica Inorganica, 6th, Puebla, Mex., Apr. 20-25, 1997 (1997), 235-238 Publisher: Asociacion Mexicana de Quimica Inorganica, Guanajuato, Mex. CODEN: 67NIAA
- DT Conference
- LA Spanish
- IT 2622-89-1, Diphenylborinic acid

RL: RCT (Reactant); RACT (Reactant or reagent)

(prepn. of tetraaza bora heterocycles derived from quanidinobenzimidazoles)

- RN 2622-89-1 CAPLUS
- CN Borinic acid, diphenyl- (6CI, 7CI, 8CI, 9CI) (CA INDEX NAME)

AB A symposium article with 3 refs. Cyclization of 2-guanidinobenzimidazole or 1-methyl-2-guanidinobenzimidazole with Ph2BOH or PhB(OH)2 in MeOH/THF

gave title compds. contg. 6-membered rings having 3 N atoms and 1 B. Structures of 2 of the PhB(OH)2 products were detd. by x-ray crystallog. RE.CNT 3 THERE ARE 3 CITED REFERENCES AVAILABLE FOR THIS RECORD

ALL CITATIONS AVAILABLE IN THE RE FORMAT

L4 ANSWER 91 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

AN 1999:205868 CAPLUS

DN 130:338147

TI Synthesis and structural characterization of (2'-hydroxyacetophenoneazine) mono(diphenylboron) chelate

AU Hopfl, H.; Farfan, N.

CS Centro Investigacione Quimicas, Univ. Autonoma del Estado Morelos, Cuernavaca, Morelos, 62210, Mex.

SO Canadian Journal of Chemistry (1998), 76(12), 1853-1859 CODEN: CJCHAG; ISSN: 0008-4042

PB National Research Council of Canada

DT Journal

LA English

IT 2622-89-1, Diphenylborinic acid

RL: RCT (Reactant); RACT (Reactant or reagent)
(prepn. and structure of hydroxyacetophenone azine diphenylboron chelates)

RN 2622-89-1 CAPLUS

CN Borinic acid, diphenyl- (6CI, 7CI, 8CI, 9CI) (CA INDEX NAME)

AB Boron chelates obtained from salicylaldehyde and 2'-hydroxyacetophenone azine are colored compds. with potential applications in anal. chem. These complexes were not studied by x-ray crystallog., although two structures with a six- or a seven-membered chelate ring are possible. The x-ray anal. of 2'-hydroxyacetophenone azine and its corresponding new mono(diphenylboron) chelate with a six-membered B heterocyclic ring are presented. With these data structural changes of the ligand on chelate formation and structural differences in comparison to salicylaldehyde azomethine B chelates are discussed.

RE.CNT 23 THERE ARE 23 CITED REFERENCES AVAILABLE FOR THIS RECORD ALL CITATIONS AVAILABLE IN THE RE FORMAT

L4 ANSWER 92 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

AN 1999:96523 CAPLUS

DN 130:168777

TI Organic compounds containing boron and aluminum, and their manufacture and use for catalysts for olefin polymerization

IN Bohnen, Hans

PA Hoechst A.-G., Germany

SO Ger. Offen., 16 pp.

CODEN: GWXXBX

DT Patent

LA German

FAN. CNT 1

PATENT NO. KIND DATE APPLICATION NO. DATE

-----PI DE 19733017 A1 19990204 DE 1997-19733017 19970731

WO				WO 1998-EP4628 19980723
			, KR, NO, US	
	RW: AT, BE, PT, SE	CH, CY	, DE, DK, ES	, FI, FR, GB, GR, IE, IT, LU, MC, NL,
	,			DE 1997-19733017A 19970731
EP	1003753 ′	A1	20000531	EP 1998-942608 19980723
EP	1003753	B1	20030305	•
	R: AT, BE,	DE, ES	, FR, GB, IT	, NL, FI
		•		DE 1997-19733017A 19970731
				WO 1998-EP4628 W 19980723
BR	9810857	Α	20000725	BR 1998-10857 19980723
				DE 1997-19733017A 19970731
				WO 1998-EP4628 W 19980723
JP	2001512128	T2	20010821	JP 2000-505172 19980723
				DE 1997-19733017A 19970731
				WO 1998-EP4628 W 19980723
AT	233771	E	20030315	AT 1998-942608 19980723
				DE 1997-19733017A 19970731
				WO 1998-EP4628 W 19980723
ZA	9806801	Α	20000131	ZA 1998-6801 19980730
				DE 1997-19733017A 19970731
US	6417302	B1	20020709	US 2000-463436 20000127
			•	DE 1997-19733017A 19970731
				WO 1998-EP4628 W 19980723
MAI	RPAT 130:1687	77		•
21	18-02-7 2622-8	39-1		
	DOM / D	> >	am /p	

os

IT

RL: RCT (Reactant); RACT (Reactant or reagent) (catalyst precursor; org. compds. contg. boron and aluminum for catalysts for olefin polymn.)

RN 2118-02-7 CAPLUS

CN Borinic acid, bis(pentafluorophenyl) - (7CI, 8CI, 9CI) (CA INDEX NAME)

RN 2622-89-1 CAPLUS

Borinic acid, diphenyl- (6CI, 7CI, 8CI, 9CI) CN (CA INDEX NAME)

AΒ R12BXAlRaRb (I; R1, Ra, Rb = H, halo, or org. group; X = group VIA element or NR; R = H or C1-20 hydrocarbyl) are manufd. by reaction of the corresponding organoaluminum compds. with the corresponding hydroxy organoborines or diorganoboric acid (anhydrides) for use with transition metal compds. as catalysts for the polymn. of olefins. I exhibit low sensitivity to catalyst poisons. A typical I was manufd. by adding a

soln. of 20 mmol bis(pentafluorophenyl)boric acid in 50 mL PhMe to 10 mmol Me3Al in PhMe at -40 degree. in 15 min and stirring 1 h each at -40.degree. and room temp.

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ANSWER 93 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN
L4
ΑN
    1999:7834 CAPLUS
DN
    130:61054
TI
    Inhibitors of .beta.-lactamases and uses therefor
ΤN
    Weston, Grady Scott; Shoichet, Brian K.
PA
    Northwestern University, USA
SO
    PCT Int. Appl., 79 pp.
    CODEN: PIXXD2
DT
    Patent
    English
LΑ
FAN.CNT 2
    PATENT NO.
                    KIND DATE
                                       APPLICATION NO. DATE
                                         _____
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PΙ
                    A1 19981217
                                       WO 1998-US12096 19980612
        W: AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CU, CZ, DE,
            DK, EE, ES, FI, GB, GE, GH, GM, GW, HU, ID, IL, IS, JP, KE, KG,
            KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MD, MG, MK, MN, MW, MX,
            NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT,
            UA, UG, UZ, VN, YU, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM
        RW: GH, GM, KE, LS, MW, SD, SZ, UG, ZW, AT, BE, CH, CY, DE, DK, ES,
            FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, BF, BJ, CF, CG, CI,
            CM, GA, GN, ML, MR, NE, SN, TD, TG
                                         US 1997-49992P P 19970613
    AU 9881408
                      A1
                           19981230
                                         AU 1998-81408
                                                         19980612
                                         US 1997-49992P P 19970613
                                         WO.1998-US12096W 19980612
                           20000621
     EP 1009415
                     Α1
                                         EP 1998-931233 19980612
        R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, NL, SE, PT, IE, FI
                                         US 1997-49992P P 19970613
                                         WO 1998-US12096W 19980612
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                                         JP 1999-503192 19980612
                           20020205
                                         US 1997-49992P P 19970613
                                         WO 1998-US12096W 19980612
PATENT FAMILY INFORMATION:
FAN 2001:91533
     PATENT NO.
                     KIND DATE
                                         APPLICATION NO. DATE
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PΙ
    US 6184363
                     B1
                           20010206
                                         US 1998-212851 19981216
                                         US 1997-49992P P 19970613
                                         US 1998-96893 A219980612
    US 6075014
                     Α
                                         US 1998-96893
                           20000613
                                                        19980612
                                         US 1997-49992P P 19970613
    US 6417174
                      В1
                           20020709
                                         US 2000-587794
                                                          20000606
                                         US 1997-49992P P 19970613
                                         US 1998-96893 A319980612
    US 6448238
                     В1
                           20020910
                                         US 2000-620268
                                                         20000719
                                         US 1997-49992P P 19970613
                                         US 1998-96893 A219980612
                                         US 1998-212851 A319981216
    MARPAT 130:61054
OS
IT
    2622-89-1, Diphenylborinic acid
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RL: BAC (Biological activity or effector, except adverse); BSU (Biological study, unclassified); PRP (Properties); THU (Therapeutic use); BIOL (Biological study); USES (Uses)

(inhibitors of .beta.-lactamases and uses in combination with .beta.-lactam antibiotics to treat resistant bacterial infections in relation to antibacterial activity and mol. modeling)

RN 2622-89-1 CAPLUS

CN Borinic acid, diphenyl- (6CI, 7CI, 8CI, 9CI) (CA INDEX NAME)

Ph | Ph— B— OH

AB The invention provides novel non-.beta.-lactam inhibitors of .beta.-lactamases. In particular, the invention provides such inhibitors which are boronic acids which is set forth in the specification. These compds. may be used with .beta.-lactam antibiotics to treat .beta.-lactam-antibiotic-resistant bacterial infections. These compds. are also antibacterial by themselves. Finally, the invention provides a pharmaceutical compn. comprising these compds.

RE.CNT 3 THERE ARE 3 CITED REFERENCES AVAILABLE FOR THIS RECORD ALL CITATIONS AVAILABLE IN THE RE FORMAT

L4 . ANSWER 94 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

AN 1998:811229 CAPLUS

DN 130:162528

TI Development of high-performance liquid chromatographic methods for measuring tetraphenylborate decomposition products in radioactive alkaline solutions

AU Hsu, Chia-lin W.; White, Thomas L.

CS Westinghouse Savannah River Technology Center, Aiken, SC, 29808, USA

SO Journal of Chromatography, A (1998), 828(1 + 2), 461-467 CODEN: JCRAEY; ISSN: 0021-9673

PB Elsevier Science B.V.

DT Journal

LA English

IT 2622-89-1, Diphenylborinic acid
RL: ANT (Analyte); FMU (Formation, unclassified); ANST (Analytical study);
FORM (Formation, nonpreparative)
 (detection in tetraphenylborate decompn. products in radioactive alk.
 solns. by HPLC)

RN 2622-89-1 CAPLUS

CN Borinic acid, diphenyl- (6CI, 7CI, 8CI, 9CI) (CA INDEX NAME)

Ph | Ph— B— OH

AB Sodium tetraphenylborate (NaTPB) was used at the Savannah River Site to ppt. and remove the gamma-emitting radionuclide Cs-137 from alk. high-level waste. The concns. of NaTPB degrdn. products such as triphenylborane, diphenylborinic acid, phenylboronic acid, and phenol indicate the rate of decompn. of TPB in storage tanks prior to processing. A simple and speedy sample prepn. protocol and two reverse-phase HPLC methods were developed to monitor the concns. of TPB and all the decompn. products in a complex matrix mainly consisting of 5 M sodium salts and 0.5 M aluminate. Approx. 4000 radioactive and nonradioactive samples per yr

were analyzed since the methods were implemented.

RE.CNT 5 THERE ARE 5 CITED REFERENCES AVAILABLE FOR THIS RECORD ALL CITATIONS AVAILABLE IN THE RE FORMAT

L4 ANSWER 95 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

AN 1998:796631 CAPLUS

DN 130:31426

TI A water adduct of tris(pentafluorophenyl)borane: (C6F5)3B(OH2)-dioxane-CH2Cl2 (1/1/1)

AU Janiak, Christoph; Braun, Lothar; Scharmann, Tobias G.; Girgsdies, Frank

CS Inst. Anorganische und Analytische Chemie, Universitat Freiburg, Freiburg, D-79104, Germany

SO Acta Crystallographica, Section C: Crystal Structure Communications (1998), C54(11), 1722-1724
CODEN: ACSCEE; ISSN: 0108-2701

PB Munksgaard International Publishers Ltd.

DT Journal

LA English

IT 216259-70-0

RL: PRP (Properties)
 (crystal structure of)

RN 216259-70-0 CAPLUS

CN Boron, aquatris(pentafluorophenyl)-, (T-4)-, compd. with dichloromethane and 1,4-dioxane (1:1:1) (9CI) (CA INDEX NAME)

CM 1

CRN 155962-45-1 CMF C18 H2 B F15 O CCI CCS

$$F \xrightarrow{F} G^{H2} \xrightarrow{F} F$$

$$F \xrightarrow{F} F$$

$$F \xrightarrow{F} F$$

CM 2

CRN 123-91-1 CMF C4 H8 O2

$$\bigcirc "$$

CM 3

CRN 75-09-2 CMF C H2 Cl2

C1-CH2-C1

AB The CH2Cl2 solvate of the 1:1 aquatris(pentafluorophenyl)borane-dioxane adduct, [B(C6F5)3(H2O)].cntdot.C4H8O2.cntdot.CH2Cl2, is a H2O complex of tris(pentafluorophenyl)borane, which was crystd. from dioxanemethylene chloride soln. Crystallog. data are given. The H2O mol. was carried in either through improperly dried solvent or during the process of crystn. The structure contains one dioxane and one CH2Cl2 mol. per formula unit. Two dioxane mols. form H bridges to the H2O mol. and also bridge between two different adduct moieties, so that an infinite chain of borane-H2O adducts and dioxane mols. is created.

RE.CNT 15 THERE ARE 15 CITED REFERENCES AVAILABLE FOR THIS RECORD ALL CITATIONS AVAILABLE IN THE RE FORMAT

L4 ANSWER 96 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

AN 1998:756217 CAPLUS

DN 130:81252

TI Diarylborinic acids as efficient catalysts for selective dehydration of aldols

AU Ishihara, Kazuaki; Kurihara, Hideki; Yamamoto, Hisashi

CS Graduate School Engineering, CREST, Japan Science Technology Corporation, Nagoya University, Nagoya, 464, Japan

SO Synlett (1997), (5), 597-599 CODEN: SYNLES; ISSN: 0936-5214

PB Georg Thieme Verlag

DT Journal

LA English

OS CASREACT 130:81252

IT 218932-40-2P, Bis(3,4,5-trifluorophenyl)borinic acid
RL: CAT (Catalyst use); SPN (Synthetic preparation); PREP (Preparation);
USES (Uses)

(catalyst for Mukaiyama aldol condensation)

RN 218932-40-2 CAPLUS

CN Borinic acid, bis(3,4,5-trifluorophenyl) - (9CI) (CA INDEX NAME)

IT 2118-02-7, Bis (pentafluorophenyl) borinic acid

RL: CAT (Catalyst use); USES (Uses)

(diarylborinates as catalysts for selective dehydration of aldols)

RN 2118-02-7 CAPLUS

CN Borinic acid, bis(pentafluorophenyl) - (7CI, 8CI, 9CI) (CA INDEX NAME)

IT 211636-23-6P

RL: CAT (Catalyst use); SPN (Synthetic preparation); PREP (Preparation); USES (Uses)

(diarylborinates as catalysts for selective dehydration of aldols)

RN 211636-23-6 CAPLUS

CN Borinic acid, bis[3,5-bis(trifluoromethyl)phenyl] - (9CI) (CA INDEX NAME)

AB Diarylborinates with electron-withdrawing substituents at their aryl groups are efficient Lewis acid catalysts for Mukaiyama aldol condensation and selective dehydration of anti-aldols to .alpha.,.beta.-enones in the presence of syn-aldols. The Lewis acidities of diarylborinates are much higher than those of the corresponding arylboronates.

RE.CNT 11 THERE ARE 11 CITED REFERENCES AVAILABLE FOR THIS RECORD ALL CITATIONS AVAILABLE IN THE RE FORMAT

L4 ANSWER 97 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

AN 1998:730205 CAPLUS

DN 130:81759

TI Stereoselective hydrolysis of p-nitrophenyl glycoside by boronic acid

AU Ohe, Takeru; Kida, Toshiyuki; Zhang, Wanbin; Nakatsuji, Yohji; Ikeda, Isao

CS Department of Applied Chemistry, Faculty of Engineering, Osaka University, Suita, 565-0871, Japan

SO Chemistry Letters (1998), (11), 1077-1078

CODEN: CMLTAG; ISSN: 0366-7022 Chemical Society of Japan

PB Chemical
DT Journal

LA English

IT 2622-89-1, Diphenylborinic acid

RL: RCT (Reactant); RACT (Reactant or reagent)

(stereoselective hydrolysis of p-nitrophenyl glycoside by boronic acid)

RN 2622-89-1 CAPLUS

CN Borinic acid, diphenyl- (6CI, 7CI, 8CI, 9CI) (CA INDEX NAME)

- AB The alk. hydrolysis of p-nitrophenyl .alpha.-D-glucoside, .alpha.-D-galactoside, and .beta.-D-mannoside was selectively accelerated by addn. of Me boronic acid, as compared to that of the corresponding .beta.-D-glucoside, .beta.-D-galactoside, and .alpha.-D-mannoside. In the case of p-nitrophenyl .alpha. (or .beta.)-D-glucoside, the .alpha./.beta. selectivity was increased up to 110 when four molar equivalents of methylor phenylboronic acid were added.
- RE.CNT 14 THERE ARE 14 CITED REFERENCES AVAILABLE FOR THIS RECORD ALL CITATIONS AVAILABLE IN THE RE FORMAT
- L4 ANSWER 98 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN
- AN 1998:725330 CAPLUS
- DN 130:110312
- TI Equilibria in the B(C6F5)3-H2O system: synthesis and crystal structures of H2O.cntdot.B(C6F5)3 and the anions [HOB(C6F5)3] and [(F5C6)3B(.mu.-OH)B(C6F5)3] -
- AU Danopoulos, Andreas A.; Galsworthy, Jane R.; Green, Malcolm L. H.; Doerrer, Linda H.; Cafferkey, Sean; Hursthouse, Michael B.
- CS Inorganic Chemistry Laboratory, Oxford, OX1 3QR, UK
- SO Chemical Communications (Cambridge) (1998), (22), 2529-2530 CODEN: CHCOFS; ISSN: 1359-7345
- PB Royal Society of Chemistry
- DT Journal
- LA English
- IT 155962-44-0P 219697-05-9P 219697-07-1P

RL: PEP (Physical, engineering or chemical process); PRP (Properties); RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); PROC (Process); RACT (Reactant or reagent)

(crystal structure; prepn., equil. and structure of)

- RN 155962-44-0 CAPLUS
- CN Boron, aquatris(pentafluorophenyl)-, dihydrate, (T-4)- (9CI) (CA INDEX NAME)

●2 H₂O

- RN 219697-05-9 CAPLUS
- CN Iridium(1+), [(1,2,5,6-.eta.)-1,5-cyclooctadiene](.eta.5-2,4-cyclopentadien-1-yl)hydro-, .mu.-hydroxyhexakis(pentafluorophenyl)diborate (1-), compd. with trichloromethane (1:2) (9CI) (CA INDEX NAME)

CM 1

CRN 67-66-3 CMF C H Cl3

CM 2

CRN 219697-04-8

CMF C36 H B2 F30 O . C13 H18 Ir

CM 3

CRN 219697-03-7 CMF C36 H B2 F30 O

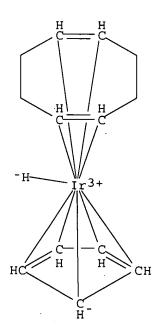
CCI CCS

$$F \longrightarrow F$$

PAGE 3-A

CM 4

CRN 63363-51-9 CMF C13 H18 Ir CCI CCS



RN 219697-07-1 CAPLUS

CN Potassium(1+), (6,7,9,10,17,18,20,21-octahydrodibenzo[b,k][1,4,7,10,13,16] hexaoxacyclooctadecin-.kappa.O5,.kappa.O8,.kappa.O11,.kappa.O16,.kappa.O19,.kappa.O22)-, (T-4)-hydroxytris(pentafluorophenyl)borate(1-), compd. with acetaldehyde and (T-4)-aquatris(pentafluorophenyl)boron (1:1:1), monohydrate (9CI) (CA INDEX NAME)

CM 1

CRN 155962-45-1 CMF C18 H2 B F15 O

Patel

9/24/2003>

CCI CCS

$$F \xrightarrow{F} C \xrightarrow{OH_2} F \xrightarrow{F} F$$

$$F \xrightarrow{F} F$$

$$F \xrightarrow{F} F$$

CM 2

CRN 75-07-0 CMF C2 H4 O

$H_3C-CH=0$

CM 3

CRN 219697-06-0 CMF C20 H24 K O6 . C18 H B F15 O

CM 4

CRN 148657-98-1 CMF C18 H B F15 O CCI CCS

$$F \longrightarrow C \xrightarrow{P} \xrightarrow{OH^{-}} F$$

$$F \longrightarrow F$$

$$F \longrightarrow F$$

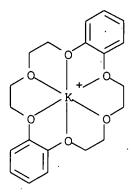
$$F \longrightarrow F$$

$$F \longrightarrow F$$

CM 5

CRN 31270-24-3

CMF C20 H24 K O6 CCI CCS



AB Addn. of water to the Lewis acid B(C6F5)3 gives the neutral compd. H2O.cntdot.B(C6F5)3.cntdot.2H2O while the reaction between B(C6F5)3 and KOH-H2O in the presence of dibenzo-18-crown-6 gives [K(dibenzo-18-crown-6)]+ [HOB(C6F5)3]- which crystallizes together with the adduct H2O.cntdot.B(C6F5)3; the new binuclear borate anion [(F5C6)3B(.mu.-OH)B(C6F5)3]- is formed as a salt with the cation [Ir(.eta.5-C5H5)(C8H12)H]+ by addn. of water to B(C6F5)3 in the presence of [Ir(.eta.5-C5H5)(C8H12)].

RE.CNT 21 THERE ARE 21 CITED REFERENCES AVAILABLE FOR THIS RECORD ALL CITATIONS AVAILABLE IN THE RE FORMAT

L4 ANSWER 99 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

AN 1998:655551 CAPLUS

DN 130:52472

TI X-ray crystallographic study of boroxazolidones obtained from L-ornithine, L-methionine, kainic acid and 2,6-pyridinedicarboxylic acid

AU Trujillo, Jose; Hopfl, Herbert; Castillo, Dolores; Santillan, Rosa; Farfan, Norberto

CS Escuela Superior de Medicina, Seccion de Graduados y Departamento de Bioquimica, Instituto Politecnico Nacional, Mexico City, CP 11340, Mex.

SO Journal of Organometallic Chemistry (1998), 571(1), 21-29 CODEN: JORCAI; ISSN: 0022-328X

PB Elsevier Science S.A.

DT Journal

LA English

IT 2622-89-1, Diphenylborinic acid
RL: RCT (Reactant); RACT (Reactant or reagent)
(reaction with kainic and pyridinedicarboxylic acid)

RN 2622-89-1 CAPLUS

CN Borinic acid, diphenyl- (6CI, 7CI, 8CI, 9CI) (CA INDEX NAME)

AB In the present contribution, the boroxazolidones prepd. from L-ornithine, L-methionine, kainic acid and 2,6-pyridinedicarboxylic acid have been studied by x-ray crystallog. A comparison of the structural data with

corresponding boroxazolidines, bicyclic boronates and tricyclic borates has shown that in boron complexes with a boroxazolidone ring the B-O bond is longer in comparison to boron complexes with a boroxazolidine ring. At the same time the N .fwdarw. B bond length is shorter indicating that the hydrolytic stability of the complexes with boroxazolidone rings is enhanced.

RE.CNT 29 THERE ARE 29 CITED REFERENCES AVAILABLE FOR THIS RECORD ALL CITATIONS AVAILABLE IN THE RE FORMAT

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L4 ANSWER 100 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN
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AN 1998:651750 CAPLUS

DN 130:20199

TI Structure-Based Enhancement of Boronic Acid-Based Inhibitors of AmpC .beta.-Lactamase

AU Weston, G. Scott; Blazquez, Jesus; Baquero, Fernando; Shoichet, Brian K.

CS Department of Molecular Pharmacology and Biological Chemistry, Northwestern University Medical School, Chicago, IL, 60611, USA

SO Journal of Medicinal Chemistry (1998), 41(23), 4577-4586 CODEN: JMCMAR; ISSN: 0022-2623

PB American Chemical Society

DT Journal

LA English

IT 2622-89-1

RL: BAC (Biological activity or effector, except adverse); BSU (Biological study, unclassified); THU (Therapeutic use); BIOL (Biological study); USES (Uses)

(structure-based enhancement of boronic acid-based inhibitors of AmpC .beta.-lactamase)

RN 2622-89-1 CAPLUS

CN Borinic acid, diphenyl- (6CI, 7CI, 8CI, 9CI) (CA INDEX NAME)

AB The expression of .beta.-lactamases is the most common form of bacterial resistance to .beta.-lactam antibiotics. To combat these enzymes, agents that inhibit (e.g. clavulanic acid) or evade (e.g. aztreonam) .beta.-lactamases have been developed. Both the .beta.-lactamase inhibitors and the .beta.-lactamase-resistant antibiotics are themselves .beta.-lactams, and bacteria have responded to these compds. by expressing variant enzymes resistant to inhibition (e.g. IRT-3) or that inactivate the .beta.-lactamase-resistant antibiotic (e.g. TEM-10). Moreover, these compds. have increased the frequency of bacteria with intrinsically resistant .beta.-lactamases (e.g. AmpC). In an effort to identify non-.beta.-lactam-based .beta.-lactamase inhibitors, we used the crystallog. structure of the m-aminophenylboronic acid-Escherichia coli AmpC .beta.-lactamase complex to suggest modifications that might enhance the affinity of boronic acid-based inhibitors for class C .beta.-lactamases. Several types of compds. were modeled into the AmpC binding site, and a total of 37 boronic acids were ultimately tested for .beta.-lactamase inhibition. The most potent of these compds., benzo[b]thiophene-2-boronic acid (36), has an affinity for E. coli AmpC of 27 nM. The wide range of functionality represented by these compds. allows for the steric and chem. "mapping" of the AmpC active site in the region of the catalytic Ser64 residue, which may be useful in subsequent

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inhibitor discovery efforts. Also, the new boronic acid-based inhibitors were found to potentiate the activity of .beta.-lactam antibiotics, such as amoxicillin and ceftazidime, against bacteria expressing class C .beta.-lactamases. This suggests that boronic acid-based compds. may serve as leads for the development of therapeutic agents for the treatment of .beta.-lactam-resistant infections.

RE.CNT 41 THERE ARE 41 CITED REFERENCES AVAILABLE FOR THIS RECORD ALL CITATIONS AVAILABLE IN THE RE FORMAT

L4 ANSWER 101 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

AN 1998:431179 CAPLUS

DN 129:188873

TI Design of Bronsted Acid-Assisted Chiral Lewis Acid (BLA) Catalysts for Highly Enantioselective Diels-Alder Reactions

AU Ishihara, Kazuaki; Kurihara, Hideki; Matsumoto, Masayuki; Yamamoto, Hisashi

CS Research Center for Advanced Waste and Emission Management (ResCWE), Nagoya University, Nagoya, 464-8603, Japan

SO Journal of the American Chemical Society (1998), 120(28), 6920-6930 CODEN: JACSAT; ISSN: 0002-7863

PB American Chemical Society

DT Journal

LA English

OS CASREACT 129:188873

IT 211636-23-6

RL: RCT (Reactant); RACT (Reactant or reagent)
(Bronsted acid-assisted chiral Lewis acid catalysts for highly enantioselective Diels-Alder reactions)

RN 211636-23-6 CAPLUS

CN Borinic acid, bis[3,5-bis(trifluoromethyl)phenyl] - (9CI) (CA INDEX NAME)

Bronsted acid-assisted chiral Lewis acids (BLA) were highly effective as chiral catalysts for the enantioselective Diels-Alder reaction of both .alpha.-substituted and .alpha.-unsubstituted .alpha., .beta.-enals with various dienes. Hydroxy groups in optically active binaphthol derivs. and boron reagents with electron-withdrawing substituents were used as Bronsted acids and Lewis acids, resp. Intramol. Bronsted acids in a chiral BLA catalyst played an important role in accelerating the rate of Diels-Alder reactions and in producing a high level of enantioselectivity. In particular, excellent enantioselectivity was achieved due to intramol. hydrogen bonding and attractive .pi.-.pi. donor-acceptor interaction in the transition-state assembly by hydroxy arom. groups in a chiral BLA catalyst.

RE.CNT 52 THERE ARE 52 CITED REFERENCES AVAILABLE FOR THIS RECORD ALL CITATIONS AVAILABLE IN THE RE FORMAT

L4 ANSWER 102 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

AN 1998:369863 CAPLUS

DN 129:136213

Patel

- TI Boron heterocycles derived from 2-guanidinobenzimidazole
- AU Andrade-Lopez, Noemi; Cartas-Rosado, Rocio; Garcia-Baez, Efren; Contreras, Rosalinda; Tlahuext, Hugo
- CS Departamento de Quimica, Centro de Investigación y de Estudios Avanzados del IPN, Mexico D.F., 07000, Mex.
- SO Heteroatom Chemistry (1998), 9(4), 399-409 CODEN: HETCE8; ISSN: 1042-7163
- PB John Wiley & Sons, Inc.
- DT Journal
- LA English
- OS CASREACT 129:136213
- RN 2622-89-1 CAPLUS
- CN Borinic acid, diphenyl- (6CI, 7CI, 8CI, 9CI) (CA INDEX NAME)

GI

AΒ The syntheses and structure detns. of a series of boron heterocycles derived from 2-guanidinobenzimidazole I (R = H, Me) are reported. Structures of new compds., 2-quanidino-1-methyl-benzimidazole (I; R = Me), diphenyl(2-guanidinobenzimidazole-N,N')borates II (R = H, Me, R1 = Ph), (hydroxy)(phenyl)(2-guanidinobenzimidazole-N,N')borates (R = H, Me, R1 =OH), (alkoxy)(phenyl)(2-guanidinobenzimidazole-N,N')borates II (R = H, R1 = MeO, Me2CHO, AcO; R = Me, R1 = MeO), dihydroxy(2-guanidino-1-methylbenzimidazole-N,N')borațe, difluoro(2-guanidinobenzimidazole-N,N')borate, dihydroxy(2-guanidino-1-benzimidazole-N,N')borate potassium salt (III), diphenyl(2-guanidinium-10H-benzimidazole-N,N')borate chloride, (methoxy) (phenyl) (2-guanidinium-10H-benzimidazole-N,N')borate chloride (IV), and N10-borane-(diphenyl-2-quanidinobenzimidazole-N,N')borate, were detd. based on 1H, 13C, 15N, and 11B spectroscopy. The x-ray diffraction structures of II (R = H, Me, R1 = Ph, OH; R = H, R1 = MeO), III, and IV were obtained. The formation of N3-borane adducts derived from I, and the dihydrido-(2-guanidinobenzimidazole-N,N')borate and dihydrido-(2-guanidino-1-methyl-benzimidazole-N,N')borate were obsd. by 11B NMR. The results

show that 2-guanidinobenzimidazole gives stable borate heterocycles with a delocalized .pi. electronic system. A dynamic exchange of N-H protons was obsd. with preferred protonation at N-12. The new heterocycles are protonated at N-10 by acidic substances to give pyridinium-type heterocycles or can lose a proton to give iminium salts.

RE.CNT 9 THERE ARE 9 CITED REFERENCES AVAILABLE FOR THIS RECORD ALL CITATIONS AVAILABLE IN THE RE FORMAT

L4 ANSWER 103 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

AN 1998:244714 CAPLUS

DN 129:27974

TI Study of cyclic borinates obtained from piperidine- and piperazine alcohols by spectroscopic methods and X-ray crystallography

AU Hopfl, Herbert; Farfan, Norberto; Castillo, Dolores; Santillan, Rosa; Gutierrez, Atilano; Daran, Jean-Claude

CS Centro de Investigaciones Quimicas, Universidad Autonoma del Estado de Morelos, Cuernavava, C.P. 62210, Mex.

SO Journal of Organometallic Chemistry (1998), 553(1-2), 221-239 CODEN: JORCAI; ISSN: 0022-328X

PB Elsevier Science S.A.

DT Journal

LA English

IT 207730-28-7P 207730-30-1P
RL: PRP (Properties); SPN (Synthetic preparation); PREP (Preparation) (prepn. and crystal structure of)

RN 207730-28-7 CAPLUS

CN Borate(1-), hydroxydiphenyl(1-piperidineethanolato-.kappa.O1)-, hydrogen, monohydrate, (T-4)- (9CI) (CA INDEX NAME)

$$\begin{array}{c|c}
\hline
OH^{-} \\
\hline
C^{-} \\
B \\
\hline
C^{-} \\
\hline
\end{array}$$

$$\begin{array}{c|c}
C^{-} \\
\hline
\end{array}$$

$$\begin{array}{c|c}
C^{-} \\
\hline
\end{array}$$

$$\begin{array}{c|c}
C^{-} \\
\hline
\end{array}$$

● H+

● H₂O

RN 207730-30-1 CAPLUS

CN Boron, hydroxydiphenyl(1-piperazineethanol-.kappa.N4)-, (T-4)-, compd. with benzene (2:1) (9CI) (CA INDEX NAME)

CM 1

CRN 207730-25-4 CMF C18 H25 B N2 O2 Page 174

10085368.2

CCI CCS

CM 2

CRN 71-43-2 CMF C6 H6



IT 207730-23-2P 207730-25-4P

RL: PRP (Properties); SPN (Synthetic preparation); PREP (Preparation) (prepn. and mol. structure of)

RN 207730-23-2 CAPLUS

CN Borate(1-), hydroxydiphenyl(1-piperidineethanolato-.kappa.Ol)-, hydrogen, (T-4)- (9CI) (CA INDEX NAME)

● н+

RN 207730-25-4 CAPLUS

CN Boron, hydroxydiphenyl(1-piperazineethanol-.kappa.N4)-, (T-4)- (9CI) (CAINDEX NAME)

IT 2622-89-1, Diphenylborinic acid

RL: RCT (Reactant); RACT (Reactant or reagent)
(prepn. of cyclic borinates obtained from piperidine- and piperazine alcs. by spectroscopic methods and x-ray crystallog.)

RN 2622-89-1 CAPLUS

CN Borinic acid, diphenyl- (6CI, 7CI, 8CI, 9CI) (CA INDEX NAME)

AB A series of eleven new 2-aminoethyl- and 3-aminopropyl borinate derivs. with a coordinative N .fwdarw. B bond has been synthesized by condensation reactions between piperidine- as well as piperazine alcs. and diphenylborinic acid. The products obtained are analogous to N-spiro compds. and bicyclic systems and have been characterized by spectroscopic methods and x-ray crystallog. Thereby the N .fwdarw. B bond and the geometry of this new heterocyclic systems have been studied in more detail.

RE.CNT 58 THERE ARE 58 CITED REFERENCES AVAILABLE FOR THIS RECORD ALL CITATIONS AVAILABLE IN THE RE FORMAT

L4 ANSWER 104 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

AN 1998:225183 CAPLUS

DN 128:316546

TI Boron(III), zinc(II), and cadmium(II) bis-chelate compounds based on tridentate pyrrolaldimines: structure and stereodynamics

AU Minkin, V. I.; Korobov, M. S.; Nivorozhkin, L. E.; Kompan, O. E.; Borodkin, G. S.; Olekhnovich, R. Ya.

CS Research Institute of Physical and Organic Chemistry, Rostov State University, Rostov-on-Don, Russia

SO Russian Journal of Coordination Chemistry (Translation of Koordinatsionnaya Khimiya) (1998), 24(3), 152-161 CODEN: RJCCEY; ISSN: 1070-3284

PB MAIK Nauka/Interperiodica Publishing

DT Journal ·

LA English

IT 205754-17-2, Phenyl-o-anisylborinic acid
RL: RCT (Reactant); RACT (Reactant or reagent)
(for prepn. of boron formylpyrrolaldiminato Ph methoxyphenyl complexes)

RN 205754-17-2 CAPLUS

CN Borinic acid, (2-methoxyphenyl)phenyl- (9CI) (CA INDEX NAME)

The dynamic 1H, 13C, and 15N NMR spectroscopic method was used to study the kinetics and mechanism of stereodynamic transformations of the 2,5-diformylpyrrolemono- and 2,5-diformylpyrroledi(N-alkylimine)-based Zn(II) and Cd(II) bis-chelate compds. and structurally similar 3,3-diaryl-1,3,2-diazaboroles in solns. X-ray diffraction anal. was performed for bis[5-cyclohexyliminoformylpyrrole-2-(N-cyclohexylaldiminato)]zinc(II) and bis[5-formylpyrrole-2-(N-isopropylaldiminato)]zinc(II). The trigonal-bipyramidal configuration of the coordination core is completed as a result of intramol. coordination by migration of the zinc between two nonequivalent sites. The tetrahedral configuration of the central metal atom in the former complex and other analogous zinc(II) and cadmium(II) complexes is found to undergo inversion through the intramol. associative mechanism.

RE.CNT 18 THERE ARE 18 CITED REFERENCES AVAILABLE FOR THIS RECORD ALL CITATIONS AVAILABLE IN THE RE FORMAT

L4 ANSWER 105 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

AN 1998:198215 CAPLUS

DN 128:181482

TI Zirconocenes as Initiators for Carbocationic Isobutene Homo- and Copolymerizations

AU Carr, Andrew G.; Dawson, David M.; Bochmann, Manfred

CS School of Chemistry, University of Leeds, Leeds, LS2 9JT, UK

SO Macromolecules (1998), 31(7), 2035-2040 CODEN: MAMOBX; ISSN: 0024-9297

PB American Chemical Society

DT Journal

LA English

IT 203399-46-6P

RL: SPN (Synthetic preparation); PREP (Preparation) (prepn. and characterization of)

RN 203399-46-6 CAPLUS

CN Zirconium, hydroxy[hydroxytris(pentafluorophenyl)borato(1-).kappa.O]bis[(1,2,3,4,5-.eta.)-1,2,3,4,5-pentamethyl-2,4-cyclopentadien-1yl]- (9CI) (CA INDEX NAME)

AB The zirconocene complexes Cp2ZrMe2 and Cp*2ZrMe2 activated with B(C6F5)3 initiate the carbocationic polymn. of isobutene and isobutene-isoprene copolymns. to IIR rubbers at temps. as high as -30 .degree.C. Unlike conventional metal halide initiators, these metallocene-based initiator systems produce both homo- and copolymers of broadly similar mol. wts. Copolymers prepd. in the presence of .apprx.2 mol% isoprene show a diene incorporation rate of 1.4-1.7%, with the typical 1,4-trans structure. Comparison of the effectiveness of zirconocene dialkyls with that of the metallocene hydrolysis products Cp*2Zr(OH)2 and (Cp2ZrMe)2(.mu.-O) in the presence of B(C6F5)3 suggests that initiation by traces of protons is less efficient than initiation by cationic metallocene alkyl species, while oxo-bridged complexes (Cp'2ZrMe)2(.mu.-O)/B(C6F5)3 (Cp' = C5H5 or C5H4SiMe3) are inactive.

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L4 ANSWER 106 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN
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AN 1998:184796 CAPLUS

DN 128:180515

TI Preparation of fungicidal cyclic boron compounds

IN Whittingham, William Guy; Lawson, Kevin Robert; Lyon, Ruth Anne

PA Zeneca Limited, UK

SO Brit. UK Pat. Appl., 28 pp. CODEN: BAXXDU

DT Patent

LA English

FAN. CNT 1

	PATENT NO.	KIND DATE		APPLICATION NO.	DATE		
ΡI	GB 2314080	A1	19971217	GB 1997-10129.	19970519		
	GB 2314080	B2	19980422				
				GB 1996-12217	19960612		

OS MARPAT 128:180515

IT 2622-89-1P, Diphenylborinic acid

RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)

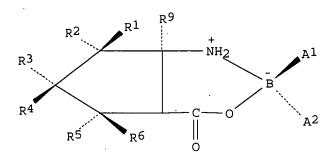
(prepn. and reaction with aminocyclopentanecarboxylic acid)

RN 2622-89-1 CAPLUS

CN Borinic acid, diphenyl- (6CI, 7CI, 8CI, 9CI) (CA INDEX NAME)



GI



Ι

The prepn. of title compds. I (A1, A2 = independently, alkyl, Ph, furyl, thienyl each optionally substituted by halogen, C1-4 alkyl, C1-4 alkoxy, C1-4 alkylthio, C1-4 haloalkyl, C1-4 haloalkoxy, C1-4 haloalkylthio; M = O, SO2, CR3R4; R1, R2, R3, R4, R5, R6 = independently H, halo, OH, C1-4 alkyl, C1-4 haloalkyl, benzyl (optionally substituted by halogen, C1-4 alkyl, C1-4 haloalkyl); R1R2 and R3R4 may together form: O,: CR7R8; R2R3 or R4R5 may together form a double bond; R2R3, R1R4, R3R5, and R4R6 may join together to form a methylene bridge; R7, R8 = independently H, halo, C1-4 alkyl; R9 = H, halo), useful as plant fungicides is described. Thus, reaction of diphenylborinic anhydride with cis-2-aminocyclopentanecarboxylic acid in Et2O gave title compd., 3,3-diphenyl-5-oxo-2-azonia-3-borata-4-oxabicyclo[4.3.0]nonane. The fungicidal activity of the compds. prepd. is given.

- L4 ANSWER 107 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN
- AN 1997:809011 CAPLUS
- DN 128:88939
- TI Syntheses and characterization of amino(pentafluorophenyl)boranes. Crystal structure of [(Me3Si)2NB(C6F5)2]
- AU Galsworthy, Jane R.; Green, Malcolm L. H.; Williams, V. Clifford; Chernega, Alex N.
- CS Inorganic Chemistry Laboratory, Oxford, OX1 3QR, UK
- SO Polyhedron (1997), Volume Date 1998, 17(1), 119-124 CODEN: PLYHDE; ISSN: 0277-5387
- PB Elsevier Science Ltd.
- DT Journal
- LA English
- IT 201163-54-4, Tetrabutylammonium fluorohydroxybis(pentafluorophenyl
)borate
 - RL: RCT (Reactant); RACT (Reactant or reagent)
 - (syntheses and characterization of amino(pentafluorophenyl)boranes)
- RN 201163-54-4 CAPLUS

Patel

CN 1-Butanaminium, N,N,N-tributyl-, (T-4)-fluorohydroxybis(pentafluorophenyl) borate(1-) (9CI) (CA INDEX NAME)

CM 1

CRN 201163-53-3 CMF C12 H B F11 O CCI CCS

CM 2

CRN 10549-76-5 CMF C16 H36 N

AB The chloroborane, ClB(C6F5)2, was studied as a useful synthon for the prepn. of amino(pentafluorophenyl)boranes. Compds. [(Me3Si)2NB(C6F5)2] (1), [(Me3Si)(H)NB(C6F5)2] and [HN{B(C6F5)2}2], were synthesized and 1 was characterized by x-ray crystallog.

RE.CNT 19 THERE ARE 19 CITED REFERENCES AVAILABLE FOR THIS RECORD ALL CITATIONS AVAILABLE IN THE RE FORMAT

L4 ANSWER 108 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

AN 1997:649938 CAPLUS

DN 127:318991

TI Dynamic NMR and X-ray diffraction study of (N-B)-diphenyl(2-aminoethoxy)borane derivatives of ephedrines and pseudoephedrines

AU Hoepfl, Herbert; Farfan, Norberto; Castillo, Dolores; Santillan, Rosa; Contreras, Rosalinda; Martinez-Martinez, Francisco Javier; Galvan, Marcelo; Alvarez, Rodolfo; Fernandez, Lilia; Halut, Sabine; Daran, Jean-Claude

CS Departamento de Quimica, Centro de Investigación y de Estudios Avanzados del IPN, Apdo. Postal 14-740, 07000, Mexico, D.F., Mex.

SO Journal of Organometallic Chemistry (1997), 544(2), 175-188 CODEN: JORCAI; ISSN: 0022-328X

PB Elsevier

DT Journal

LA English

OS CASREACT 127:318991

IT 2622-89-1, Diphenylborinic acid

RL: RCT (Reactant); RACT (Reactant or reagent)
 (prepn., crystal structure and dynamic NMR of (N-B)-diphenyl(2 aminoethoxy)borane derivs. of ephedrines and pseudoephedrines)

RN 2622-89-1 CAPLUS

CN Borinic acid, diphenyl- (6CI, 7CI, 8CI, 9CI) (CA INDEX NAME)

GI

The prepn. and characterization of various (N-B)-diphenyl-(2-AΒ aminoethoxy) boranes (e.g. I) derived from ephedrine and pseudoephedrine derivs. are reported: (N-B)-diphenyl(1-(R)-phenyl-2-(S)-methyl-2aminoethoxy)borane (1b), (N-B)-diphenyl(1-(R)-phenyl-2-(R)-methyl-2aminoethoxy) borane (2b), (N-B)-diphenyl [N-(R)-methyl-(1-(R)-phenyl-2-(S)methyl-2-aminoethoxy)]borane (3b-trans), (N-B)-diphenyl[N-(S)-methyl-(1-(R)-phenyl-2-(S)-methyl-2-aminoethoxy)]borane (3b-cis), (N-B) -diphenyl (N-(S) -methyl - (1-(R) -phenyl - 2-(R) -methyl - 2-(R)aminoethoxy)]borane (4b-trans), (N-B)-diphenyl[N,N-dimethyl-(1-(R)-phenyl-2-(S)-methyl-2-aminoethoxy)]borane (5b) and (N-B)-diphenyl[N,N-dimethyl-(1-(R)-phenyl-2-(R)-methyl-2-aminoethoxy)]borane (6b). The five membered N.fwdarw.B cyclic structures 1b-6b were assigned based on 1H-, 13C-, 11Band 15N-NMR data and all compds. except for 3b-trans were subjected to x-ray diffraction anal. showing N.fwdarw.B bond lengths of 1.66(2) and 1.64(2) .ANG. for 1b, 1.657(9) and 1.664(9) .ANG. for 2b, 1.68(2) .ANG. for 3b-cis, 1.66(1) .ANG. for 4b-trans, 1.744(8) .ANG. for 5b and 1.74(1) .ANG. for 6b. The study of the intramol. N.fwdarw.B coordination by dynamic NMR spectroscopy afforded .DELTA.G.dbldag. values of 67.9, 70.9, 64.8, 68.2, 49.7 and 52.7 kJ mol-1 for the dissocn. of the N.fwdarw.B bond in compds. 1b-6b resp. Steric interactions between the substituents at the (2-aminoethoxy)borane ring det. the stability of the N.fwdarw.B bond as well as the N configuration. Theor. calcns. of the electrostatic charges for the B and N atoms in 1b, 2b, 3b-cis, 3b-trans, 4b-cis, 5b and 6b show that the increase of pos. charge on the N atom causes a shift to lower frequencies in the 15N NMR spectra.

- L4 ANSWER 109 OF 309 · CAPLUS COPYRIGHT 2003 ACS on STN
- AN 1997:576714 CAPLUS
- DN 127:209854
- TI Treatment of wastewater for removal of organoboron compounds
- IN Reimer, Ronald Anthony
- PA E.I. Du Pont De Nemours and Co., USA
- SO PCT Int. Appl., 11 pp.

CODEN: PIXXD2 DTPatent LΑ English FAN.CNT 1 PATENT NO. KIND DATE APPLICATION NO. DATE ------____ _____ . _____ PΙ WO 9730759 A1 19970828 WO 1997-US2255 19970212 W: CA, CN, JP, SG RW: AT, BE, CH, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE US 1996-606140 A 19960223 US 5709841 Α 19980120 US 1996-606140 19960223 CA 2244948 AA 19970828 CA 1997-2244948 19970212 CA 2244948 C 20010821 US 1996-606140 A 19960223 A1 19981209 EP 881924 EP 1997-905939 19970212 EP 881924 В1 20010523 R: DE, ES, FR, GB, NL US 1996-606140 A 19960223 WO 1997-US2255 W 19970212 CN 1211931 Α 19990324 CN 1997-192479 19970212 CN 1103613 В 20030326 US 1996-606140 A 19960223 JP 11504262 T2 JP 1997-530224 19970212 19990420 JP 3285878 B2 20020527 US 1996-606140 A 19960223 WO 1997-US2255 W 19970212 ES 2158500 Т3 20010901 ES 1997-905939 19970212 US 1996-606140 A 19960223 IT 2622-89-1, Diphenylborinic acid RL: POL (Pollutant); REM (Removal or disposal); OCCU (Occurrence); PROC (Process) (treatment of wastewater for removal of organoboron compds.) 2622-89-1 CAPLUS RN

Ph | Ph— B— OH

CN

AB An aq. soln. of organoboron compds. is hydrolyzed to boric acid and the corresponding org. compd. by treatment at >150.degree. and a pressure sufficient to prevent substantial evapn., and at pH of 5-9.

L4 ANSWER 110 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

Borinic acid, diphenyl- (6CI, 7CI, 8CI, 9CI)

AN 1997:558880 CAPLUS

DN . 127:176003

TI Bis(pentafluorophenyl)borinic Acid as a Highly Effective Oppenauer Oxidation Catalyst for Allylic and Benzylic Alcohols

AU Ishihara, Kazuaki; Kurihara, Hideki; Yamamoto, Hisashi

CS Graduate School of Engineering, Nagoya University, Nagoya, 464-01, Japan

SO Journal of Organic Chemistry (1997), 62(17), 5664-5665 CODEN: JOCEAH; ISSN: 0022-3263

PB American Chemical Society

DT Journal

LA English

OS CASREACT 127:176003

(CA INDEX NAME)

IT 194089-33-3 194089-34-4

RL: CAT (Catalyst use); USES (Uses) (bis(pentafluorophenyl)borinic acid effective Oppenauer oxidn. catalyst for allylic and benzylic alcs.)

RN 194089-33-3 CAPLUS

CN Borinic acid, bis[2,4,6-trifluoro-3,5-bis(trifluoromethyl)phenyl]- (9CI) (CA INDEX NAME)

RN 194089-34-4 CAPLUS

CN Borinic acid, bis[2,6-difluoro-3,4,5-tris(trifluoromethyl)phenyl]- (9CI) (CA INDEX NAME)

IT 2118-02-7

RL: CAT (Catalyst use); PEP (Physical, engineering or chemical process); RCT (Reactant); PROC (Process); RACT (Reactant or reagent); USES (Uses) (bis(pentafluorophenyl)borinic acid effective Oppenauer oxidn. catalyst for allylic and benzylic alcs.)

RN 2118-02-7 CAPLUS

CN Borinic acid, bis(pentafluorophenyl) - (7CI, 8CI, 9CI) (CA INDEX NAME)

AB The Oppenauer (OPP) oxidn. is one of the most useful method for transforming from secondary alcs. into ketones. In general, however, it is difficult to oxidize primary alcs. to the corresponding aldehydes by the OPP method. We report here that bis(pentafluorophenyl)borinic acid

(1) is a suitable OPP catalyst for primary and secondary allylic and benzylic alcs. The Lewis acidity of 1 is stronger than pentafluorophenylboronic acid. Catalysis was carried out using 1 to 2 mol% of 1 loading in the reaction of allylic alcs. in the presence of 3 equiv of pivalaldehyde as a hydride acceptor and 1 equiv of magnesium sulfate as a drying reagent at ambient temp. Mechanistically, the present reaction is similar to the ordinary OPP oxidns. catalyzed by metal alkoxides, but, in contrast to the basic nature of these catalysts, the driving force of the oxidn. in this case is the activation of pivalaldehyde by the strong Lewis acid 1.

L4 ANSWER 111 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

AN 1997:429061 CAPLUS

DN 127:176456

TI Synthesis and structure of 2,2'-boryl-, germyl-, silyl-, and stannyl-substituted 1,1'-binaphthyl systems

AU Schilling, Birgit; Kaiser, Volker; Kaufmann, Dieter E.

CS Inst. Organische Chemie, Techn. Univ. Clausthal, Clausthal-Zellerfeld, D-38678, Germany

SO Chemische Berichte/Recueil (1997), 130(7), 923-932 CODEN: CHBRFW

PB Wiley-VCH

DT Journal

LA English

OS CASREACT 127:176456

IT 194038-55-6P

RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)

(prepn. and structure of boryl-, germyl-, silyl-, and stannylbinaphthalenes)

RN 194038-55-6 CAPLUS

CN Borinic acid, bis([1,1'-binaphthalen]-2-yl)-, stereoisomer (9CI) (CA INDEX NAME)

GΙ

I

A no. of Lewis acid binaphthyl systems, substituted in 2- or AB 2,2'-position, were synthesized by lithiation of 2,2'-dibromo-1,1'binaphthyl, I (R, R1 = Br) followed by addn. of various electrophiles. Stepwise lithiation and subsequent borylation with B(OMe)3 leads to the bromo boronate I [R = B(OH)2, R1 = Br], which is stabilized by esterification with pinacol. By increasing the reaction mixt. to 2 equiv BuLi and 2 equiv B(OMe) 3 the path to the binaphthyl monoboronate I [R = BR22, R1 = H, R22 = O(CMe2)20] is opened up. A further increase in the quantity of electrophile also leads to the binaphthyl bisboronates I [R, R1 = BR22; R22 = O(CMe2)20]. The 2,2'-disubstituted silyl, germyl, and stannyl derivs. I (R = R1 = SiMe3, GeMe3, SnMe3) are accessible in good yields. Treatment with B halides leads exclusively to Me/halo exchange, given the bidentate Lewis acids I (R = R1 = SiBr3, GeCl3, SnCl3), the former of which can be bridged by O. Only in the case of bis(tributylstannyl)binaphthyl I (R, R1 = SnBu3), ipso substitution occurs in the presence of BCl3, giving the bis(dichloroboryl)binaphthyl I (R, R1 = BCl2) which can then be hydrolyzed to the diboronate I [R, R1 = B(OH)2]. The structures of the majority of compds. were studied by x-ray diffraction. In case of I (R = R1 = SiMe3, GeMe3, SnMe3), the naphthyl groups are oriented perpendicular to each other. Intra- and intermol. interactions are dominated by this binaphthyl system. In the case of the O-bridged compds. I [RR1 = SiBr2OSiBr2, GeCl2OGeCl2], the angle between the naphthyl planes decreases to .apprx.70.degree.. This also affects the packing of the mol. In this instance the orientation of 2 naphthyl groups in neighboring mols. is nearly parallel. The structure of the diboronates is dominated by intra- and intermol. H bonding.

L4 ANSWER 112 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

AN 1997:381760 CAPLUS

DN 127:121799

TI Synthesis and structural characterization of some novel metalloboroxides bearing boron-bound mesityl and fluoromesityl substituents: the molecular structure of the first metallaboroxane complex

AU Gibson, Vernon C.; Redshaw, Carl; Clegg, William; Elsegood, Mark R. J.

CS Dep. Chem., Imperial Coll., South Kensington, London, SW7 2AY, UK

SO Polyhedron (1997), 16(15), 2637-2641 CODEN: PLYHDE; ISSN: 0277-5387

PB Elsevier

DT Journal

LA English

IT 192823-38-4P

RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)

(prepn. and metalation reaction with lithium and molybdenum)

RN 192823-38-4 CAPLUS

CN Borinic acid, bis[2,4,6-tris(trifluoromethyl)phenyl] - (9CI) (CA INDEX

NAME)

IT 192823-43-1

RL: RCT (Reactant); RACT (Reactant or reagent)
 (reaction with copper dibromide)

RN 192823-43-1 CAPLUS

CN Borinic acid, bis[2,4,6-tris(trifluoromethyl)phenyl]-, lithium salt (9CI) (CA INDEX NAME)

● Li

Treatment of the new boronous acid HOB(fmes)2 (1) (fmes = 2,4,6-(CF3)3C6H2) with BuLi in Et2O/pentane affords, after work-up, the dimer [Li(THF)OB(fmes)2]2 (2). Reaction of [Mo2(NMe2)6] with two equiv. of 1 in toluene gives the amido-boroxide complex Mo2(NMe2)4[OB(fmes)2]2 (3). Treatment of CuBr2 with LiOB(mes)2 (mes = 2,4,6-Me3C6H2) in THF affords after work-up and addn. of excess pyridine the monomeric Cu(II) boroxide, {Cu[O3B2(mes)2]2[Li(MeCN)(C5H5N)]2} (4), contg. the new ligand mesB(O)OB(O)mes, as a result of loss of mesitylene and formation of a B-O-B bond. 2 And 4 were structurally characterized by x-ray crystallog.

- L4 ANSWER 113 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN
- AN 1997:198358 CAPLUS
- DN 126:293511
- TI Competitive transport of reducing sugars through a lipophilic membrane facilitated by aryl boron acids
- AU Karpa, Michael J.; Duggan, Peter J.; Griffin, Gregory J.; Freudigmann, Stacy J.
- CS Sch. Mol. Sciences, James Cook Univ. North Queensland, Townsville, 4811, Australia
- SO Tetrahedron (1997), 53(10), 3669-3678 CODEN: TETRAB; ISSN: 0040-4020
- PB Elsevier
- DT Journal
- LA English
- IT 2622-89-1

RL: RCT (Reactant); RACT (Reactant or reagent)

(competitive transport of reducing sugars through a lipophilic membrane

facilitated by aryl boron acids)

RN 2622-89-1 CAPLUS

CN Borinic acid, diphenyl- (6CI, 7CI, 8CI, 9CI) (CA INDEX NAME)

Ph | Ph— B— OH

AB The extn. and competitive transport of fructose, glucose, and sucrose through dichloroethane facilitated by combinations of aryl boron acids and tetraalkyl ammonium salts is described. Although extn. abilities of the boron acids are comparable, there are distinct differences in their sugar transport properties. The aq. soly. of the aryl boron acid is apparently a crit. controlling factor of sugar flux. A combination of PBA and aliquat 336 in the membrane effectively separates fructose from an equimolar mixt. of fructose, glucose and sucrose, despite extn. expts. displaying comparable extn. of glucose and fructose.

L4 ANSWER 114 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

AN 1997:84568 CAPLUS

DN 126:86119

TI Solutions containing triphenylborane-octadecylamine complex as antifouling agents for fish nets

IN Usu, Toshihiro; Shimada, Akira; Shibuya, Keiji; Katsuyama, Kazuki

PA Yoshitomi Pharmaceutical, Japan

SO Jpn. Kokai Tokkyo Koho, 12 pp.

CODEN: JKXXAF

DT Patent

LA Japanese

FAN.CNT 2

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE		
PI	JP 08295609	A2	19961112	JP 1996-81861	19960227		
				JP 1995-37968	19950227		

PATENT FAMILY INFORMATION:

FAN 1997:48814

	PATENT NO.		DATE	APPLICATION NO.	DATE		
ΡI	JP 08295608	A2	19961112	JP 1995-305520	19951124		
	JP 2978749	B2	19991115				
				JP 1994-289483	19941124		
				JP 1995-37968	19950227		

OS MARPAT 126:86119

IT 12113-07-4

RL: RCT (Reactant); RACT (Reactant or reagent) (solns. contg. triphenylborane-octadecylamine complex as antifouling

agents for fish nets)

RN 12113-07-4 CAPLUS

CN Borate(1-), hydroxytriphenyl-, sodium, (T-4)- (9CI) (CA INDEX NAME)

$$C = B = C$$

● Na+

AB Antifouling agents for fish nets comprise solns. consisting of Ph3B.NH2Rl (I; Rl = octadecyl) and org. solvents. Octadecylamine was treated with Ph3B.NaOH at 70.degree. for 3 h to give 90% I. I 10, LV 50 (polybutene) 7.5, yellow vaseline 7.5, acrylic resin 20, and xylene 55% were mixed to prep. an antifouling agent, which was applied to a Tetoron fish net to show no attachment of hydrozoan after storage in seawater for 4 mo.

L4 ANSWER 115 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

AN 1997:76760 CAPLUS

DN 126:89553

TI Electrophilic Methylplatinum Complexes: First Structure of a Hydroxytris(pentafluorophenyl)borate Complex

AU Hill, Geoffrey S.; Manojlovic-Muir, Ljubica; Muir, Kenneth W.; Puddephatt, Richard J.

CS Departments of Chemistry, University of Western Ontario, London, ON, N6A 5B7, Can.

SO Organometallics (1997), 16(4), 525-530 CODEN: ORGND7; ISSN: 0276-7333

PB American Chemical Society

DT Journal

LA English

OS CASREACT 126:89553

IT 185555-10-6P

RL: PRP (Properties); SPN (Synthetic preparation); PREP (Preparation) (prepn. and crystal structure of)

RN 185555-10-6 CAPLUS

CN Platinum, [4,4'-bis(1,1-dimethylethyl)-2,2'-bipyridine-.kappa.N1,.kappa.N1'] [hydroxytris(pentafluorophenyl)borato(1-)-

.kappa.O]methyl-, (SP-4-2)- (9CI) (CA INDEX NAME)

IT 185555-07-1P 185555-08-2P 185555-09-3P

RL: SPN (Synthetic preparation); PREP (Preparation)

(prepn. of)

RN

185555-07-1 CAPLUS
Platinum(1+), [4,4'-bis(1,1-dimethylethyl)-2,2'-bipyridine-CN · .kappa.N1,.kappa.N1']carbonylmethyl-, (SP-4-3)-, (T-4)hydroxytris(pentafluorophenyl)borate(1-) (9CI) (CA INDEX NAME)

CM1

CRN 177593-40-7 C20 H27 N2 O Pt CMF

CCI CCS

CM 2

CRN 148657-98-1 CMF C18 H B F15 O

CÇI CCS

RN 185555-08-2 CAPLUS

CN Platinum(1+), [4,4'-bis(1,1-dimethylethyl)-2,2'-bipyridine-.kappa.N1,.kappa.N1'](.eta.2-ethene)methyl-, (T-4)-hydroxytris(pentafluorophenyl)borate(1-) (9CI) (CA INDEX NAME)

CM 1

CRN 177593-43-0 CMF C21 H31 N2 Pt CCI CCS

CM 2

CRN 148657-98-1 CMF C18 H B F15 O CCI CCS

$$F \longrightarrow C^{-} \longrightarrow B^{3+} \longrightarrow F$$

$$F \longrightarrow F$$

$$F \longrightarrow F$$

$$F \longrightarrow F$$

$$F \longrightarrow F$$

RN 185555-09-3 CAPLUS

CN Platinum(1+), [4,4'-bis(1,1-dimethylethyl)-2,2'-bipyridine-.kappa.N1,.kappa.N1']methyl(triphenylphosphine)-, (SP-4-2)-, (T-4)-hydroxytris(pentafluorophenyl)borate(1-) (9CI) (CA INDEX NAME)

CM 1

CRN 185555-05-9 CMF C37 H42 N2 P Pt CCI CCS

CM 2

CRN 148657-98-1 CMF C18 H B F15 O CCI CCS

$$F \longrightarrow F \longrightarrow F \longrightarrow F \longrightarrow F$$

GΙ

Patel

Ι

Ι

AB Treatment of [PtMe2(t-Bu2bpy)] (t-Bu2bpy = 4,4'-di-tert-butyl-2,2'bipyridine) with B(C6F5)3 in the presence of the ligand L gives, under anhyd. conditions, [PtMeL(t-Bu2bpy)][MeB(C6F5)3] {L = Co (1a), C2H4 (2a), PPh3 (3a) }. Similar reactions performed in the presence of H2O afford $[PtMeL(t-Bu2bpy)][HOB(C6F5)3] \{L = CO(1b), C2H4(2b), PPh3(3b)\}.$ In the absence of L, the treatment of [PtMe2(t-Bu2bpy)] with B(C6F5)3 and H2O gives [Pt{HOB(C6F5)3}Me(t-Bu2bpy)] (4) (shown as structure I), the 1st published example of a hydroxytris(pentafluorophenyl)borate complex. All of the complexes are fully characterized by NMR and IR spectroscopy and, in the case of complex 4, by an x-ray anal. which confirms the attachment of the [HOB(C6F5)3] - ligand to the square-planar Pt atom via a Pt-O bond of 2.062(2) .ANG.. Probably in the presence of H2O, B(C6F5)3 forms an adduct [H2OB(C6F5)3] (which acts as a strong acid H[HOB(C6F5)3]) and this then protonates a Pt-Me bond of [PtMe2(t-Bu2bpy)] forming CH4 and [PtMe(t-Bu2bpy)]+[HOB(C6F5)3]-. This protonolysis methodol. provides an alternative route to the well-established electrophilic Me-ligand abstraction by B(C6F5)3 for the prodn. of late transition-metal cations.

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L4 ANSWER 116 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN
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AN 1997:56168 CAPLUS

DN 126:89565

TI Method for producing triphenylborane-amine complexes

IN Usu, Toshihiro; Shimada, Akira; Shibuya, Keiji; Katsuyama, Kazuki

PA Yoshitomi Pharmaceutical, Japan

SO Jpn. Kokai Tokkyo Koho, 6 pp.

CODEN: JKXXAF

DT Patent

LA Japanese

FAN.CNT.1

	O212. = 1						
PATENT NO.		KIND	DATE	APPLICATION NO.	DATE		
ΡI	JP 08311074	A2	19961126	JP 1996-52908	19960311		
				.TP 1995-55507	19950315		

OS MARPAT 126:89565

IT 12113-07-4 185421-90-3

RL: RCT (Reactant); RACT (Reactant or reagent) (prepn. of triphenylborane-amine complexes by reaction of

triphenylborane-sodium or potassium hydroxide adduct with amines)

RN 12113-07-4 CAPLUS

CN Borate(1-), hydroxytriphenyl-, sodium, (T-4)- (9CI) (CA INDEX NAME)

Patel

● Na+

RN 185421-90-3. CAPLUS
CN Borate(1-), hydroxytriphenyl-, potassium, (T-4)- (9CI) (CA INDEX NAME)

● K+

GI

$$\begin{bmatrix} & & & & & & \\ & & & & & \\ & & & & & \\ & & & & & \\ & & & & & \\ & & & & & \\ & & & & \\ & & & & \\ & & & & \\ & & & & \\ & & & & \\ & & &$$

AB The title compds. (I; X = Q, Q1; wherein R2, R3, R4 = alkyl, haloalkyl, cycloalkyl, Ph, halo, alkoxy, alkenyl, aralkyl) are prepd. by reaction of triphenylborane-sodium or potassium hydroxide adduct Ph3B+-OH.Na+ or

Ph3B+-OH.K+ with R1NH2 or pyridine deriv. (II; R2 - R4 = same as above) in an aq. soln. This process is industrially advantageous, since it does not use expensive and very flammable ether but it uses water as the solvent and can be carried out in the air without fire hazard. Moreover the starting material, triphenylborane-sodium or potassium hydroxide adduct, is stable and safely handled. These compds. I are useful as antifouling agents for fishing nets, coatings for bottom of ships, industrial preservatives and antifungal agents, or animal repellents (no data). Thus, 10.8 g n-propylamine was slowly added dropwise over 30 min at 20.degree. to a 9.6% aq. soln. of Ph3B+-OH.Na+ (461.0 g) under stirring, upon which crystals immediately pptd., and the resulting mixt. was allowed to react at 20.degree. for 3 h to give, after filtering off the crystals and washing them with water, 90% triphenylborane-n-propylamine complex.

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ANSWER 117 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN
L4
     1996:685037 CAPLUS
AN
     126:18813
DN
     Twofold ring cleavage of 2,3,5,6-tetrahydro-10bH-oxazolo[2,3-
TI
     a]isoquinoline with salts of hydroxylamine derivatives
ΑU
     Moehrle, H.: Tot, E.: Steiner, S.
     Inst. Pharm. Chem., Heinrich-Heine-Univ., Duesseldorf, D-40225, Germany
CS
SO
     Journal fuer Praktische Chemie/Chemiker-Zeitung (1996), 338(8), 711-717
     CODEN: JPCCEM; ISSN: 0941-1216
PB
     Barth
DT
     Journal
LΑ
     German
OS
     CASREACT 126:18813
IT
     2622-89-1, Diphenylborinic acid
     RL: RCT (Reactant); RACT (Reactant or reagent)
        (ring cleavage of tetrahydrooxazoloisoquinoline with hydroxylammonium
        derivs.)
     2622-89-1 CAPLUS
RN
     Borinic acid, diphenyl- (6CI, 7CI, 8CI, 9CI) (CA INDEX NAME)
CN
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Ph
|-
Ph— B— OH
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IT 184167-71-3P

RL: SPN (Synthetic preparation); PREP (Preparation) (ring cleavage of tetrahydrooxazoloisoquinoline with hydroxylammonium derivs.)

RN 184167-71-3 CAPLUS

CN Borinic acid, diphenyl-, compd. with 2,3,6,10b-tetrahydro-5H-oxazolo[2,3-a]isoquinoline (1:1) (9CI) (CA INDEX NAME)

CM 1

CRN 95071-86-6 CMF Cl1 H13 N O

CM 2

CRN 2622-89-1 CMF C12 H11 B O

GI

AB The reaction of the title compd. (I) with HONH2.HCl led to the ring-opened [(hydroxyethyl)aminoethyl]benzaldehyde oxime II (R = OH). Similar products II (R = OMe, OCH2CH:CH2, OCMe3, OCH2Ph, OCPh3, NHCONH2, NHCONHPh) were available from appropriate hydroxylamine ether and semicarbazide salts. The reactivity of I and the stability of the products was investigated.

L4 ANSWER 118 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

AN 1996:657110 CAPLUS

DN 126:3408

TI Selective Monosaccharide Transport through Lipid Bilayers Using Boronic Acid Carriers

AU Westmark, Pamela R.; Gardiner, Stephen J.; Smith, Bradley D.

ΙI

CS Department of Chemistry and Biochemistry, University of Notre Dame, Notre Dame, IN, 46556, USA

SO Journal of the American Chemical Society (1996), 118(45), 11093-11100 CODEN: JACSAT; ISSN: 0002-7863

PB American Chemical Society

DT Journal

LA English

IT 2622-89-1, Diphenylborinic acid

RL: NUU (Other use, unclassified); PEP (Physical, engineering or chemical process); PROC (Process); USES (Uses)

(selective monosaccharide transport through lipid bilayers using boronic acid carriers)

RN 2622-89-1 CAPLUS

CN Borinic acid, diphenyl- (6CI, 7CI, 8CI, 9CI) (CA INDEX NAME)

Patel

Ph | Ph— B— OH

ΑB Twenty-one boronic acids were studied for their ability to transport saccharides in and out of liposomes. The rates of liposome efflux were detd. using an enzymic assay, whereas the influx studies used a radiotracer method. All boronic acids examd., except those that were highly hydrophilic, facilitated monosaccharide transport. The order of transport selectivity was sorbitol > fructose > glucose. The disaccharides maltose and sucrose were not transported to any significant degree. Facilitated transport was demonstrated with a variety of liposome types, including multilamellar and unilamellar vesicles with anionic or cationic polar lipid additives. Transport mechanism studies included the accumulation of structure-activity data, as well as systematic investigations of various environmental changes such as pH, added salt, membrane potential, and temp. Overall, the evidence is strongly in favor of a membrane carrier mechanism. The boronic acid combines reversibly with a diol group on the monosaccharide to produce a tetrahedral, anionic boronate, which is the major complexed structure in bulk, aq. soln. the bilayer surface, the tetrahedral boronate is in equil. with its neutral, trigonal form, which is the actual transported species. At low carrier concns., a first-order dependence on carrier was obsd. indicating that the transported species was a 1:1 sugar-boronate. At higher carrier concns. the kinetic order approached 2, suggesting the increased participation of a 1:2 sugar-bisboronate transport pathway. The effect of boronic acids on liposomal bilayer fluidity was probed by fluorescence spectroscopy using appropriate reporter mols. Adding cholesterol to the liposome membranes reduced translational fluidity by "packing and ordering" the bilayer. Addn. of lipophilic arylboronic acids (either free or complexed with monosaccharides) induced a similar but smaller effect.

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L4
    ANSWER 119 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN
AN
     1996:649665 CAPLUS
DN
     125:276888
TI
     Tris(pentafluorophenyl)borate complexes and olefin polymerization
     catalysts derived from them
IN
     Siedle, Allen R.; Miller, John A.; Lamanna, William M.
PA
     Minnesota Mining and Mfg. Co., USA
     PCT Int. Appl., 45 pp.
SO
     CODEN: PIXXD2
DT
     Patent
LΑ
     English
FAN.CNT 1
     PATENT NO.
                      KIND
                            DATE
                                           APPLICATION NO.
                                                             DATE
                      ----
                            -----
PΙ
     WO 9626967
                            19960906
                                                            19960119
                      A1
                                           WO 1996-US737
        W: AL, AM, AT, AU, AZ, BB, BG, BR, BY, CA, CH, CN, CZ, DE, DK, EE,
             ES, FI, GB, GE, HU, IS, JP, KE, KG, KP, KR, KZ, LK, LR, LS, LT,
             LU, LV, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE,
             SG, SI
```

RW: KE, LS, MW, SD, SZ, UG, AT, BE, CH, DE, DK, ES, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, BF, BJ, CF, CG, CI, CM, GA, GN, ML, MR,

US 1995-396966 19950301

Patel

NE. SN

AU	9647027	A1	19960918	AU	1996-47027	19960119
				US	1995-396966	19950301
				WO	1996-US737	. 19960119
EP	812335	. A1	19971217	EP	1996-902730	19960119
	R: DE,	FR; GB, IT	, NL			
				US	1995-396966	19950301
				WO	1996-US737	19960119
EP	891991	A2	19990120	EP	1998-118324	19960119
EP	891991	A3	19990224			
	R: DE,	FR, GB, IT	, NL			
				US	1995-396966	19950301
				EP	1996-902730	19960119
JP	11501342	T2	19990202	· JP	1996-526244	19960119
				US	1995-396966	19950301
				WO	1996-US737	19960119

IT 147892-18-0P

RL: CAT (Catalyst use); IMF (Industrial manufacture); PREP (Preparation); USES (Uses)

(tris(pentafluorophenyl)borate complexes and olefin polymn. catalysts derived from them)

RN 147892-18-0 CAPLUS

CN Borate(1-), hydroxytris(pentafluorophenyl)-, (T-4)-, hydrogen, compd. with N,N-diethylethanamine (1:1) (9CI) (CA INDEX NAME)

CM 1

CRN 147892-17-9 CMF C18 H B F15 O . H CCI CCS

● H+

CM 2

CRN 121-44-8 CMF C6 H15 N

Tris(pentafluorophenyl)borane complexes having general formula (C6F5)Bx(YXH)q [X = 0, S; q = 1-3; Y = H, C1-500 hydrocarbyl which may 0 and/or F, R13Si, (R2)2C:N; R1 = C1-25 alkyl, Ph, SiO-contg. group; R2 = C1-25 hydrocarbyl] are synthesized and used in combination with other organometallic compds. as catalysts for polymn. and copolymn. of olefins. Rubbery polymers produced by using these catalysts are useful in making pressure-sensitive adhesives and packaging film. Propylene was polymd. by using catalysts including tris(pentafluorophenyl)borane complex with octadecanol, [1,2-bis(9-fluorenyl)ethane]zirconium di-Me, and tri-n-octylaluminum to give a polymer with wt.-av. mol. wt. 500,000, no.-av. mol. wt. 228,000, polydispersity index 2.59, and Tg 270 K.

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L4 ANSWER 120 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN
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AN 1996:532251 CAPLUS

DN 125:212798

TI SPE-HPLC determination of catecholamines using an affinity principle

AU Brandsteterova, E.; Kubalec, P.; Krajnak, K.; Skacani, I.

CS Department Analytical Chemistry, Slovak Technical University, Bratislava, 812 37, Slovakia

SO Neoplasma (1996), 43(2), 107-112 CODEN: NEOLA4; ISSN: 0028-2685

PB Slovak Academic Press

DT Journal

LA English

IT 2622-89-1

RL: ARG (Analytical reagent use); ANST (Analytical study); USES (Uses) (SPE-HPLC detn. of catecholamines in human urine)

RN 2622-89-1 CAPLUS

CN Borinic acid, diphenyl- (6CI, 7CI, 8CI, 9CI) (CA INDEX NAME)

Solid-phase extn. (SPE) with the affinity chromatog. principle was applied AB for high performance liq. chromatog. (HPLC) detn. of catecholamines in patients urine samples. Electrochem. amperometric detection (ED) was used for all HPLC analyses and both anal. and microbore columns were tested for optimal chromatog. resoln. of all analyzed catecholamines. Capacity ratio k' and detection limits were evaluated for all columns used. Precolumn affinity chromatog. procedure of catecholamines with diphenylboronic acid (DPBA) in alk. medium improved their retention on different com. SPE reversed-phase cartridges. After clean-up and preconcn. step the complex catecholamine-diphenylboronic acid was degraded by acidic pH and eluted from SPE cartridge. After SPE affinity step catecholamines were analyzed by HPLC-ED. The optimal elution solns. was chosen also as suitable SPE cartridges. Extn. recoveries of catecholamines were 89.6-93.3% with relative std. deviation RSD = 3.8-4.3%. Total anal. time was 30 min including 15 min for the presepn. procedure.

L4 ANSWER 121 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

AN 1996:321015 CAPLUS DN 124:343648 Heterocyclylalkyl diarylboron ester and thioester fungicidal agents ΤI Patel, Bomi Pilloo; Van Tuyl Cotter, Henry; Lavanish, Jerome Michael ΙN PA American Cyanamid Company, USA SO Eur. Pat. Appl., 17 pp. CODEN: EPXXDW DT Patent LΑ English FAN.CNT 1 PATENT NO. KIND DATE APPLICATION NO. DATE ______ EP 1995-306682 PIEP 703235 A2 19960327 19950921 EP 703235 Α3 19980107 R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IE, IT, LI, LU, MC, NL, PT, SE US 1994-313525 19940926 US 5591726 19970107 US 1994-313525 19940926 IL 115401 IL 1995-115401 19950922 A1 19990411 US 1994-313525 19940926 HU 71992 A2 · 19960328 HU 1995-2796 19950925 HU 216441 В 19990628 US 1994-313525 19940926 AU 1995-32845 AU 9532845 Α1 19950925 19960404 AU 704406 B2 19990422 US 1994-313525 19940926 BR 9504166 Α 19960806 BR 1995-4166 19950925 US 1994-313525 19940926 JP 08245644 19960924 JP 1995-268954 Α2 19950925 US 1994-313525 19940926 CN 1129062 Α 19960821 CN 1995-116053 19950926 US 1994-313525 19940926 MARPAT 124:343648 OS ΙT 2622-89-1 176913-70-5

RL: RCT (Reactant); RACT (Reactant or reagent)

(prepn. of heterocyclylphenylalkyl diphenylborinates from)

2622-89-1 CAPLUS RN

CN Borinic acid, diphenyl- (6CI, 7CI, 8CI, 9CI) (CA INDEX NAME)

RN 176913-70-5 CAPLUS

CN Borinic acid, bis(4-fluorophenyl) - (9CI) (CA INDEX NAME)

GΙ

$$F \xrightarrow{B} \xrightarrow{N} F$$

$$C1 \xrightarrow{C1}$$

AB The present invention provides heterocyclylalkyl diarylboron ester and thioester compds., e.g., I, and their use for the prevention, control or amelioration of diseases caused by fungi (test data given). Further provided are compns. and methods comprising those compds. for the protection of plants from fungal infection and disease.

L4 ANSWER 122 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

Ι

AN 1996:409 CAPLUS

DN 124:175135

TI Synthesis and Characterization of Organic Materials with Conveniently Accessible Supercooled Liquid and Glassy Phases: Isomeric 1,3,5-Tris(naphthyl)benzenes

AU Whitaker, Craig M.; McMahon, Robert J.

CS Department of Chemistry, University of Wisconsin, Madison, WI, 53706-1396, USA

SO Journal of Physical Chemistry (1996), 100(3), 1081-90 CODEN: JPCHAX; ISSN: 0022-3654

PB American Chemical Society

DT Journal

LA English

IT 62981-91-3P

RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)

(prepn. of tris(naphthyl)benzenes with supercooled liq. and glassy phases)

RN 62981-91-3 CAPLUS

CN Borinic acid, di-1-naphthalenyl- (9CI) (CA INDEX NAME)

AB 1,3,5-Tris(1-naphthyl)benzene [i.e., 1,1',1''-(1,3,5-benzenetriyl)tris[naphthalene]], 1,3-bis(1-naphthyl)-5-(2-naphthyl)benzene and 1-(1-naphthyl)-3,5-bis(2-naphthyl)benzene easily supercool, and form glasses on cooling from the melt. The above compds. were prepd. using Suzuki's conditions to effect the cross-coupling reaction of 1,3,5-tribromobenzene with 1-naphthylboronic acid and/or 2-naphthylboronic

acid. Variable-temp. 13C NMR studies of 1,3,5-tris(1-naphthyl)benzene establish a barrier of ca. 12 kcal/mol for rotation about the aryl-aryl bond; this value displays good agreement with the barrier of 13 kcal/mol computed using mol. mechanics calcns. (MM2). This relatively low rotational barrier is inconsistent with the previously-held notion that 1,3,5-tris(1-naphthyl)benzene exists as a mixt. of noninterconverting rotational isomers (atropisomers) in soln. at room temp. Variable-temp. 13C NMR studies of 1,3-bis(1-naphthyl)-5-(2-naphthyl)benzene establish barriers of ca. 12 kcal/mol for rotation about the 1-naphthyl-aryl bond and <9 kcal/mol for rotation about the 2-naphthyl-aryl bond. Again, these values display good agreement with the barriers of 14 kcal/mol and 2 kcal/mol computed using mol. mechanics calcns. (MM2). Earlier syntheses of 1,3,5-tris(1-naphthyl)benzene provided materials that were poorly characterized by modern stds. 1H and 13C NMR spectra of one such material, a sample widely-used in studies of glasses and supercooled ligs., establish the structure of the material as 1,3-bis(1-naphthyl)-5-(2naphthyl)benzene, not 1,3,5-tris(1-naphthyl)benzene. This revised structural assignment necessitates a re-evaluation of the earlier literature. Differential scanning calorimetry (DSC) establishes that tris(naphthyl)benzenes melt (Tm = 182 .degree.C, 194 .degree.C and 147 .degree.C, resp.) and form glasses upon cooling (Tg = 81 .degree.C, 77 .degree.C and 67 .degree.C, resp.). Given its low m.p. and glass transition temp., 1-(1-naphthyl)-3,5-bis(2-naphthyl)benzene is an attractive candidate for future studies of mol. dynamics of glassy materials.

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ANSWER 123 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN
L4
AN
      1995:526887 CAPLUS
DN
      122:258671
ΤI
      Wood preservatives containing boronic, diboronic, or borinic acids
ΙN
      Lloyd, Jeffrey Douglas; Deane-Wray, Allison Kathleen
PA
      U.S. Borax Inc., USA
SO
      Brit. UK Pat. Appl., 18 pp.
      CODEN: BAXXDU
DT
      Patent
      English
LA
FAN CNT 1
      PATENT NO.
                           KIND DATE
                                                     APPLICATION NO. DATE
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                                  _____
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PΙ
      GB 2281210
                            A1
                                   19950301
                                                     GB 1993-17297
                                                                           19930819
                                                     WO 1994-GB1810 19940818
      WO 9505081
                            Α1
                                  19950223
          W: AM, AT, AU, BB, BG, BR, BY, CA, CH, CN, CZ, DE, DK, ES, FI, GB, GE, HU, JP, KE, KG, KP, KR, KZ, LK, LT, LU, LV, MD, MG, MN, MW, NL, NO, NZ, PL, PT, RO, RU, SD, SE, SI, SK, TJ, TT, UA, US, UZ, VN RW: KE, MW, SD, AT, BE, CH, DE, DK, ES, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, BF, BJ, CF, CG, CI, CM, GA, GN, ML, MR, NE, SN, TD, TG
                                                     GB 1993-17297
                                                                           19930819
                                                                        19940818
      AU 9473902
                            A1
                                   19950314
                                                     AU 1994-73902
                                                     GB 1993-17297
                                                                           19930819
                                                     WO 1994-GB1810
                                                                           19940818
      MARPAT 122:258671
OS
      2622-89-1P, Diphenylborinic acid 71173-48-3P
IΤ
      RL: BUU (Biological use, unclassified); IMF (Industrial manufacture); SPN
      (Synthetic preparation); BIOL (Biological study); PREP (Preparation); USES
          (prepn. as wood preservative)
RN
      2622-89-1 CAPLUS
      Borinic acid, diphenyl- (6CI, 7CI, 8CI, 9CI) (CA INDEX NAME)
CN
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RN 71173-48-3 CAPLUS

CN Borinic acid, diphenyl-, compd. with 2-aminoethanol (1:1) (9CI) (CA INDEX NAME)

CM 1

CRN 2622-89-1 CMF C12 H11 B O

CM 2

CRN 141-43-5 CMF C2 H7 N O

 $H_2N-CH_2-CH_2-OH$

AB A liq. biocidal or preservative compn. which is enzyme free comprises (i) as active ingredient at least one borinic, boronic or diboronic acid, (ii) at least one surfactant, and (iii) an aq. or org. solvent or carrier and is highly effective in protecting porous substrates, particularly cellulosic substrates such as timber, from attack by fungi, bacteria or insects. The active ingredient is at least one organoboron compd. of formula R1(R2)nB[(R2)mR3]OH, R1(R2)nB[(R2)mOH]OH, or HOB[(R2)mOH](R2)nR4(R2)nR4(R2)nB[(R2)mOH]OH, where R1 and R3 = optionally substituted alkyl, cycloalkyl, aralkyl, or aryl (C1-20), and R2 and R4 = -[CH(X)]o-, -[C(X)=C(X)]p-, or -[CH(X)]q- where X=H, optionally substituted C1-6 alkyl, aryl, OH, halogen, amino, nitro, thiol, aldehyde, carboxylic acid, sulfonate or phosphorate or their derivs. or salts, and where o, p, and q = 0, 1, or 2, and m and n = 0 or 1. Thus, 0.1% phenylboronic acid applied to filter papers killed subterranean termites (Reticulotermes santonensis) in 96-120 h and was more active than the std. wood preservative sodium octaborate. Phenylboronic acid and its halogenated derivs. were likewise active against Coniophora puteana and Coriolus versicolor.

- L4 ANSWER 124 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN
- AN 1995:444247 CAPLUS
- DN 122:213862
- TI Preparation of sesamol
- IN Kumamoto, Nobumitsu; Uchibori, Yukitaka; Umeno, Masayuki
- PA Hokko Chem Ind Co, Japan
- SO Jpn. Kokai Tokkyo Koho, 5 pp.

CODEN: JKXXAF

DT Patent LA Japanese

FAN.CNT 1

	C111 1						
	PATENT NO.	KIND DATE		APPLICATION NO.	DATE		
ΡI	JP 06345756	A2	19941220	JP 1993-156387	19930603		
	JP 2614812	B2	19970528				
				JP 1993-156387	19930603		

OS CASREACT 122:213862; MARPAT 122:213862

IT 161800-66-4P

RL: IMF (Industrial manufacture); RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent) (prepn. of sesamol)

RN 161800-66-4 CAPLUS

CN Borinic acid, bis(1,3-benzodioxol-5-yl)- (9CI) (CA INDEX NAME)

GΙ

AB Sesamol (I) is prepd. in several steps from benzenemagnesium halide II [X = halo]. Thus, reaction of II [X = Br] with tri-Bu borate, followed by treatment with aq. 10% sulfuric acid soln., and reaction with H2O2, gave 80.2% I.

L4 ANSWER 125 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

AN 1995:298350 CAPLUS

ΙI

DN 122:265465

TI Tris(2,6-dimethoxyphenyl)borane and its adducts

AU Wada, Masanori; Kanzaki, Mitsuyuki; Ogura, Hideo; Hayase, Shuichi; Erabi,
Tatsuo

CS Department of Materials Science, Faculty of Engineering, Tottori University, Koyama, Tottori, 680, Japan

SO Journal of Organometallic Chemistry (1995), 485(1-2), 127-33 CODEN: JORCAI; ISSN: 0022-328X

PB Elsevier

DT Journal

LA English

IT 162719-59-7P

RL: PRP (Properties); SPN (Synthetic preparation); PREP (Preparation) (prepn. and properties of tris(2,6-dimethoxyphenyl)borane adducts and tris(2,6-dimethoxyphenyl)cyanoborates)

RN 162719-59-7 CAPLUS

CN Borate(1-), tris(2,6-dimethoxyphenyl)hydroxy-, lithium, (T-4)- (9CI) (CA INDEX NAME)

• Li+

AB Tris(2,6-dimethoxyphenyl)borane, .PHI.3B (.PHI. = 2,6-(MeO)2C6H3), an air-stable cryst. compd., formed isolable 1 : 1 adducts with NH3 and some primary amines but not with tertiary amines, secondary amines, or sec-alkylamines. Some tetraalkylammonium tris(2,6-dimethoxyphenyl)cyanoborates [R4N][.PHI.3BCN] are sol. both in H2O and in benzene, and the stearyltrimethylammonium salt is sol. even in n-hexane. The f.p. depression measurements for some tetraalkylammonium salts suggested that they are assocd. in benzene by several mols.

L4 ANSWER 126 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

AN 1995:262746 CAPLUS

DN 122:239778

TI Hindered organoboron groups in organic chemistry. 28. The solvolyses of bis(2,6-dimethyl-4-methoxyphenyl)organylboranes, (DMP)2BR

AU Pelter, Andrew; Drake, Robert

CS Department Chemistry, University College Swansea, Swansea, SA2 8PP, UK

SO Tetrahedron (1994), 50(48), 13801-28 CODEN: TETRAB; ISSN: 0040-4020

PB Elsevier

DT Journal

LA English

IT 50481-12-4

RL: RCT (Reactant); RACT (Reactant or reagent) (prepn. of 4-methoxy-2,6-dimethylphenol from)

RN 50481-12-4 CAPLUS

CN Borinic acid, bis(4-methoxy-2,6-dimethylphenyl)- (9CI) (CA INDEX NAME)

AB Mineral acid catalyzed methanolysis of bis(2,6-dimethyl-4-methoxyphenyl)organylboranes, (DMP)2BR, is much faster than that of the corresponding dimesitylorganylboranes, Mes2BR. This allows for the release of organyl groups from overhindered boranes. It also provides a link between (DMP)2BR, from which .alpha.-carbanions can be produced, and RB(OMe)2 which do not yield .alpha.-carbanions. Solvolyses can be enhanced using glycol, which renders even HOAc an effective solvolysis catalyst.

Page 204

- L4 ANSWER 127 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN
- AN 1995:262745 CAPLUS
- DN 122:239777
- TI Hindered organoboron groups on organic chemistry. 27. Preparations and some properties of alkylbis(2,6-dimethyl-4-methoxyphenyl)boranes ((DMP)2BR)
- AU Pelter, Andrew; Drake, Robert
- CS Department Chemistry, University College Swansea, Swansea, SA2 8PP, UK
- SO Tetrahedron (1994), 50(48), 13775-800 CODEN: TETRAB: ISSN: 0040-4020
- PB Elsevier
- DT Journal
- LA English
- OS CASREACT 122:239777
- IT 50481-12-4P
 - RL: SPN (Synthetic preparation); PREP (Preparation)
 (prepns. and properties of alkylbis(2,6-dimethyl-4methoxyphenyl)boranes)
- RN 50481-12-4 CAPLUS
- CN Borinic acid, bis(4-methoxy-2,6-dimethylphenyl)- (9CI) (CA INDEX NAME)

- AB Alkylbis(2,6-dimethyl-4-methoxyphenyl)boranes ((DMP)2BR) were synthesized in an attempt to overcome the limitations of the steric hindrance approach to the prodn. of B stabilized carbanions. Anion prodn. from (DMP)2BR,followed by alkylations and condensations with aldehydes are reported. Redn. of (DMP)2BF with K hydride yields the corresponding hydroborate. Attempts to isolate (DMP)2BH were unsuccessful but the borane was readily trapped with alkynes, yielding alkenylboranes. The allyl deriv., (DMP)2BAllyl, was made and some of its reactions were studied.
- L4 ANSWER 128 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN
- AN 1995:205961 CAPLUS
- DN 122:3588
- TI Diaryl (pyridinio and isoquinolinio) boron insecticidal and acaricidal agents
- IN Patel, Bomi P.
- PA American Cyanamid Co., USA
- SO U.S., 7 pp. CODEN: USXXAM

DT LA FAN.	Patent English CNT 2		
	PATENT NO.	KIND DATE	APPLICATION NO. DATE
PI	US 5354741 EP 624315 EP 624315	A 19941011 A1 19941111 B1 19980701	US 1993-59143 19930507 EP 1994-106124 19940420
	R: AT, BE,	CH, DE, DK, ES,	FR, GB, GR, IE, IT, LI, LU, NL, PT, SE US 1993-59048 A 19930507
	AT 167781	E 19980715	US 1993-59048 A 19930507
	ES 2118281	T3 19980916	US 1993-59143 A 19930507 ES 1994-106124 19940420 US 1993-59048 A 19930507
	CZ ⁻ 287713	B6 20010117	US 1993-59143 A 19930507 CZ 1994-982 19940422 US 1993-59048 A 19930507
	JP 07138265	A2 19950530	US 1993-59143 A 19930507
	IL 109506	A1 19990817	US 1993-59143 A 19930507 IL 1994-109506 19940502
	CA 2122954	AA 19941108	US 1993-59048 A 19930507 US 1993-59143 A 19930507 CA 1994-2122954 19940505
			US 1993-59048 A 19930507 US 1993-59143 A 19930507
	BR 9401889	A 19941129	BR 1994-1889 19940505 US 1993-59048 A 19930507 US 1993-59143 A 19930507
	AU 9461951 AU 677295	A1 19941110 B2 19970417	
	ZA 9403172	A 19950111	US 1993-59143 A 19930507 ZA 1994-3172 19940506
	HU 67803	A2 19950529	US 1993-59048 A 19930507 US 1993-59143 A 19930507 HU 1994-1439 19940506
		• .	US 1993-59048 A 19930507 US 1993-59143 A 19930507
	PL 178746	B1 20000630	PL 1994-303339 19940506 US 1993-59048 A 19930507 US 1993-59143 A 19930507
	CN 1099557 CN 1071995	A 19950308 B 20011003	CN 1994-105225 19940507
PATE	NT FAMILY INFORM	ATION:	US 1993-59048 A 19930507 US 1993-59143 A 19930507
FAN	1995:205960 PATENT NO.	KIND DATE	APPLICATION NO. DATE
PI	EP 624315 EP 624315	A 19941011 A1 19941111 B1 19980701	US 1993-59048 19930507 EP 1994-106124 19940420
	R: AT, BE,	CH, DE, DK, ES,	FR, GB, GR, IE, IT, LI, LU, NL, PT, SE US 1993-59048 A 19930507

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			•		1993-59143	Α	19930507
JP	07138265	A2	19950530		1994-115829		19940502
					1993-59048	Α	19930507
				US	1993-59143	Α	19930507
ΙL	109506	A1	19990817	IL	1994-109506		19940502
				US	1993-59048	Α	19930507
		•		US	1993-59143	Α	19930507
CA	2122954	AA	19941108	CA	1994-2122954		19940505
					1993-59048		19930507
					1993-59143	Α	19930507
BR	9401889	A	19941129		1994-1889		19940505
					1993-59048		19930507
					1993-59143	А	19930507
	9461951	A1	19941110	ΑU	1994-61951		19940506
AU	677295	B2	19970417				
	•				1993-59048		19930507
					1993-59143	Α	19930507
ZA	9403172	A	19950111		1994-3172		19940506
					1993-59048		19930507
					1993-59143	A	19930507
HU	67803	A2	19950529		1994-1439		19940506
					1993-59048		19930507
	4-4-14				1993-59143	A	19930507
ЪГ	178746	B1	20000630		1994-303339		19940506
					1993-59048		19930507
~~	1000555	_			1993-59143	Α	19930507
	1099557	A	19950308	CN	1994-105225		1994.0507
CN	1071995	В	20011003			_	
					1993-59048		19930507
	177020		10061006		1993-59143	A	19930507
IN	177039	A	19961026		1994-CA346	_	19940510
				US	1993-59048	A	19930507

OS MARPAT 122:3588 '

RL: BAC (Biological activity or effector, except adverse); BSU (Biological study, unclassified); SPN (Synthetic preparation); BIOL (Biological study); PREP (Preparation)

(prepn. and insecticidal and acaricidal activity of boron derivs.)

IT 159565-82-9P

RN .159565-82-9 CAPLUS

CN Boron, (3-butylpyridine)hydroxydiphenyl-, (T-4)- (9CI) (CA INDEX NAME)

IT 2622-89-1, Diphenylborinic acid

RL: RCT (Reactant); RACT (Reactant or reagent) (reaction with butylpyridine)

RN 2622-89-1 CAPLUS

Borinic acid, diphenyl- (6CI, 7CI, 8CI, 9CI) (CA INDEX NAME) CN

GI

$$\begin{array}{c|c}
X & & & \\
C - - B + 3 - C - & \\
N & & \\
R1 & & \\
R2 & & \\
\end{array}$$

AΒ Insecticidal and acaricidal diaryl (pyridinio and isoquinolinio) boron compds. having the structure (I; where Xm = X and Yn = Y are H, halo, alkyl, haloalkyl, alkoxy, etc. with m and n = 0, 1, 2 or 3; R = alkyl, alkoxy, halo, hydroxy; R1 - R3 = H, halo, alkyl, haloalkyl, alkoxy, haloalkoxy, cyano, nitro, etc.) were prepd. and used for the protection of plants from attack by insects and acarina.

ANSWER 129 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN L4

Ι

AN1995:205960 CAPLUS

DN 122:125925

Diaryl (pyridinio and isoquinolinio) boron fungicidal agents TI

ΙN Patel, Bomi P.

PΑ American Cyanamid Co., USA

SO U.S., 9 pp.

CODEN: USXXAM Patent

DT LΑ

English

FAN.CNT 2

PATENT NO. KIND DATE APPLICATION NO.

Patel

R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IE, IT, LI, LU, NL, PT, SE	PI	ΕP	5354740 624315 624315		A A1 B1	19941011 19941117 19980701		US EP	1993-59048 1994-10612	1	19930507 19940420		
AT 167781 E 19980715 AT 1994-106124 19940420 US 1993-59048 A 19930507 US 1993-59048 A 1			R: AT			, DK, ES,	FR,	GB, (GR, IE, IT,	LI	, LU, NL,	PT,	SE
AT 167781 E 19980715 AT 1994-106124 19940420 US 1993-59143 A 19930507 US 1993-59048 A 19930507 U								US	1993-59048	Α	19930507		
S 1933-59048 A 1930507 US 1933-59048 A 1930507								US	1993-59143	Α	19930507		
ES 2118281 T3 19980916 ES 1994-106124 19940420 US 1993-59048 A 19930507 CZ 287713 B6 20010117 CZ 1993-59048 A 19930507 US 1993-59143 A 19930507 US 1993-59048 A 19930507 US 1993-59048 A 19930507 US 1993-59048 A 19930507 US 1993-59048 A 19930507 US 1993-59143 A 19930507 US 1993-59048 A 19930507		ΑT	167781		E	19980715		AT	1994-10612	1	19940420		
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CZ 287713 B6 20010117 CZ 1994-982 19940422 US 1993-59048 A 19930507 US 1993-59143 A 19930507 US 1993-59048 A 19930507 US 1993-59143 A 19930507 US 1993-59048 A 19930507 US		ES	2118281		Т3	19980916		ES	1994-106124	1	19940420		
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IL 109506		JΡ	0713826	5	A2	19950530		JP	1994-115829	9	19940502		
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OD 1333-03040 A 1333050/			_										

					1993-59143	A	19930507
ES	2118281	Т3	19980916	ES	1994-106124		19940420
				US	1993-59048	Α	19930507
				US	1993-59143	Α	19930507
CZ	287713	В6	20010117	CZ	1994-982		19940422
				US	1993-59048	Α	19930507
			•	US	1993-59143	Α	19930507
JΡ	07138265	A2	19950530	JP	1994-115829		19940502
	•			US	1993-59048	A	19930507
				US	1993-59143		19930507
IL	109506	A1	19990817	ΙL	1994-109506		19940502
				US	1993-59048	Α	19930507
					1993-59143		19930507
CA	2122954	AΑ	19941108	CA	1994-2122954		19940505
				US	1993-59048	Α	19930507
					1993-59143		19930507
BR	9401889	Α	19941129	BR	1994-1889		19940505
					1993-59048	Α	19930507
				US	1993-59143		19930507
AU	9461951	A1	19941110	AU	1994-61951		19940506
ΑU	677295	B2	19970417				
				US	1993-59048	Α	19930507
					1993-59143		19930507
ZA	9403172	Α	19950111		1994-3172		19940506
					1993-59048	Α	19930507
					1993-59143		19930507
HU	67803	A2	19950529		1994-1439		19940506
					1993-59048	Α	19930507
					1993-59143		19930507
$_{ m PL}$	178746	B1	20000630	PL	1994-303339		19940506
				US	1993-59048	Α	19930507
					1993-59143		19930507
CN	1099557	A	19950308		1994-105225		19940507
CN	1071995	В	20011003				
				US	1993-59048	Α	19930507
				US	1993-59143	Α	19930507

OS MARPAT 122:125925

IT 159565-82-9P 161098-82-4P

RL: BAC (Biological activity or effector, except adverse); BSU (Biological study, unclassified); SPN (Synthetic preparation); BIOL (Biological study); PREP (Preparation)

(diaryl (pyridinio and isoquinolinio) boron fungicidal agents)

RN 159565-82-9 CAPLUS

CN Boron, (3-butylpyridine)hydroxydiphenyl-, (T-4)- (9CI) (CA INDEX NAME)

RN 161098-82-4 CAPLUS

CN Boron, hydroxy[4-(1-methylethyl)pyridine]diphenyl-, (T-4)- (9CI) (CA

INDEX NAME)

IT 2622-89-1, Diphenylborinic acid RL: RCT (Reactant); RACT (Reactant or reagent)

(reaction with butylpyridine)

RN 2622-89-1 CAPLUS

CN Borinic acid, diphenyl- (6CI, 7CI, 8CI, 9CI) (CA INDEX NAME)

GΙ

$$\begin{array}{c|c}
X & & & \\
C^{-} - B^{+3} - C^{-} & & \\
N & & & \\
R^{1} & & & \\
R^{2} & & & \\
\end{array}$$

Diaryl (pyridinio and isoquinolinio) boron compds. having the structural formula I (Xm = X and Yn = Y are H, halo, alkyl, haloalkyl, etc.; m and n = 0, 1, 2, or 3; R = alkyl, alkoxy, halo or hydroxy; R1, R2, R3 = H, alkyl, haloalkyl, alkoxy, etc.) are used for the prevention, control or amelioration of diseases caused by phytopathogenic fungi. Compns. and methods comprising those compds. for the protection of plants from fungal infestation and disease are described.

L4 ANSWER 130 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

Ι

- AN 1995:202653 CAPLUS
- DN 122:285282
- TI Molecular recognition with boron acids. 8. Diphenylborinic acid is a strong inhibitor of serine proteases
- AU Steiner, Steven J.; Bien, Jeffrey T.; Smith, Bradley D.
- CS Dep. Chem. Biochem., Univ. Notre Dame, Notre Dame, IN, 46556, USA
- SO Bioorganic & Medicinal Chemistry Letters (1994), 4(20), 2417-20

CODEN: BMCLE8; ISSN: 0960-894X

PB Elsevier

DT Journal

LA English

IT 2622-89-1, Diphenylborinic acid

RL: BAC (Biological activity or effector, except adverse); BSU (Biological study, unclassified); BIOL (Biological study)

(diphenylborinic acid as strong inhibitor of serine proteases)

RN 2622-89-1 CAPLUS

CN Borinic acid, diphenyl- (6CI, 7CI, 8CI, 9CI) (CA INDEX NAME)

Ph | Ph— B— OH

AB Diphenylborinic acid, a com. available and reasonably air-stable compd., was found to be a strong competitive inhibitor of 3 serine proteases. Compared to phenylboronic acid, it was a 30-fold better inhibitor of alpha.-chymotrypsin, a 15-fold better inhibitor of subtilisin BPN', and a 60-fold better inhibitor of bovine trypsin. The pKa and inhibitory ability of methylphenylborinic acid was also detd.

L4 ANSWER 131 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

AN 1995:55046 CAPLUS

DN 122:3693

TI Transport of Glycosides through Liquid Organic Membranes Mediated by Reversible Boronate Formation is a Diffusion-Controlled Process

AU Morin, Gregory T.; Hughes, Martin Patrick; Paugam, Marie-France; Smith, Bradley D.

CS Department of Chemistry and Biochemistry, University of Notre Dame, Notre Dame, IN, 46556, USA

SO Journal of the American Chemical Society (1994), 116(20), 8895-901 CODEN: JACSAT; ISSN: 0002-7863

DT Journal

LA English

IT 2622-89-1, Diphenylborinic acid

RL: BSU (Biological study, unclassified); PEP (Physical, engineering or chemical process); PRP (Properties); BIOL (Biological study); PROC (Process)

(transport of glycosides through liq. org. membranes mediated by reversible boronate formation is a diffusion-controlled process)

RN 2622-89-1 CAPLUS

CN Borinic acid, diphenyl- (6CI, 7CI, 8CI, 9CI) (CA INDEX NAME)

Ph | Ph— B— OH

The ability of phenylboronic acid, [3-(1-adamantylcarboxamido)phenyl]boron ic acid, and diphenylborinic acid to ext. and transport p-nitrophenyl .beta.-D-glucopyranoside (glucoside), p-nitrophenyl .beta.-D-galactopyranoside (galactoside), and p-nitrophenyl .beta.-D-mannopyranoside (mannoside) through a liq org membrane, in the presence of trioctylmethylammonium or tetrabutylammonium chloride, was detd. Under

the conditions examd., glycoside transport was facilitated by the reversible formation of covalent tetrahedral, anionic glycoside-boronate complexes, which partitioned into the org. membrane as lipophilic ion pairs. The results of various expts. indicated the rate-limiting step in the transport process was diffusion of the solutes through the narrow unstirred boundary layers adjacent the org./aq. interfaces. A plot of glycoside transport rate vs. glycoside extn. const., Kex, formed an approx. bell-shaped relation. Maximal transport occurred when the carrier admixt. had an extn. const. of log Kex(max) apprx. 2.2. Under low extn. conditions (Kex < Kex(max)), movement of the glycoside from the receiving phase into the org. membrane was the rate-detg. step, and under high extn. conditions (Kex > Kex(max)), exit from the membrane into the receiving phase was rate-detg. Because transport was dependent on Kex, an anal. of the structural and environmental factors that controlled transport could be reduced to an anal. of the factors that changed Kex relative to Kex(max). The factors examd. included the following; pH, boron acid acidity, diol structure, polarity of the org. layer, boron acid lipophilicity, glycoside lipophilicity, quaternary ammonium lipophilicity, and the presence of competing lipophilic anions. The importance of Kex(max) as the parameter detg. transport stereoselectivity is discussed.

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L4 ANSWER 132 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN AN 1994:695090 CAPLUS
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DN 121:295090

TI Diarylboron ester and thioester fungicidal agents

IN Patel, Bomi P.

PA American Cyanamid Co., USA

SO U.S., 8 pp. CODEN: USXXAM

DT Patent

LA English

FAN.CNT 1

PATENT NO. KIND DAT	TE API	PLICATION NO.	DATE
PI US 5348947 A 199			19930507 19930507

OS MARPAT 121:295090

IT 89566-59-6

RL: RCT (Reactant); RACT (Reactant or reagent)
 (reaction with hydroxyethylpyridine)

RN 89566-59-6 CAPLUS

CN Borinic acid, bis(4-chlorophenyl) - (9CI) (CA INDEX NAME)

IT 2622-89-1, Diphenylborinic acid

RL: RCT (Reactant); RACT (Reactant or reagent) (reaction with pyridinemethanol or quinaldic acid)

RN 2622-89-1 CAPLUS

CN Borinic acid, diphenyl- (6CI, 7CI, 8CI, 9CI) (CA INDEX NAME)

GI

Diarylboron esters and thioesters having the structural formula (I, X = Xm and Y = Yn = H, halogen, C1-C6 alkoxy, C1-C6 haloalkoxy, etc., n = integer of 0, 1, 2 or 3; A = O or S; R-R3 = hydrogen, halogen, C1-C4 alkoxy, C1-C4 haloalkoxy, etc.; R4 and R5 = H, halogen, C1-C8 alkoxy, etc.; W = (CH2)p, where p = integer of 0 or 1) are fungicides and were prepd. by reacting a substituted pyridine with diarylborinic acid. These boron fungicides are effective against phytopathogenic fungi causing apple scab, downy mildew, rice blast, powdery mildew, etc.

L4 ANSWER 133 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

Ι

AN 1994:695089 CAPLUS

DN 121:295089

TI 2,2-diaryl-1-(oxa.and thia)-2a-azonia-2-borataacenaphthene fungicidal

IN Patel, Bomi P.

PA American Cyanamid Co., USA

SO U.S., 8 pp.
CODEN: USXXAM

DT Patent

LA English

FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
ΡI	US 5348948	 А	19940920	US 1993-59940	19930507
				US 1993-59940	19930507

OS MARPAT 121:295089

IT 66117-64-4

RN 66117-64-4 CAPLUS

CN Borinic acid, bis(4-methylphenyl) - (9CI) (CA INDEX NAME)

GI

AB 2,2-Diaryl-1-(oxa and thia)-2a-azonia-2-borataacenaphthene compds. having the structural formula (I, A = O or S; R - R5 = H, halo, alkoxy, etc.; X and Y = H, halo, etc.) are fungicides which could be prepd. by reacting a substituted quinoline with diarylborinic acid. Compns. and methods comprising those compds. for the protection of plants from fungal infestation and disease are described.

L4 ANSWER 134 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

Ι

AN 1994:630821 CAPLUS

DN 121:230821

TI Structural studies of organoboron compounds. LXI. Synthesis of (alkylideniminoxy)diarylboranes and crystal and molecular structure of dimeric (ethylideniminoxy)bis(4-methoxyphenyl)borane [3,3,6,6-tetrakis(4-methoxyphenyl)-2,5-diethylidene-1,4-dioxa-2,5-diazonia-3,6-diboratacyclohexane]

AU Kliegel, Wolfgang; Nanninga, Dierk; Riebe, Ulf; Rettig, Steven J.; Trotter, James

CS Inst. Pharma. Chem., Tech. Univ. Braunschweig, Braunschweig, 38106, Germany

SO Canadian Journal of Chemistry (1994), 72(7), 1735-40 CODEN: CJCHAG; ISSN: 0008-4042

DT Journal

LA English

RN 73774-45-5 CAPLUS

CN Borinic acid, bis(4-methoxyphenyl) - (9CI) (CA INDEX NAME)

GΙ

Oxime diarylborinates, e.g., I, were obtained from several aliph. and arom. aldoximes as well as from cyclic ketoximes by acylation with a diarylborinic acid or anhydride (R2BX, R = aryl, X = OH or OBR2). These compds. could also be synthesized by condensation of an (aminoxy)diarylborane, which supposedly has a cyclodimeric BONBON structure, with an aldehyde or with a ketone. An x-ray anal. of I was carried out. A dimeric structure featuring a central six-membered BONBON ring was established. This represents the first structurally characterized example of a monocyclic BONBON ring.

L4 ANSWER 135 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

AN 1994:624863 CAPLUS

DN 121:224863

TI Boronic acids as enzyme models: Catalysis of oxime formation

Ι

AU Niu, Ling-Hao; Frias, Bernarda; Sharma, Subodh; Philipp, Manfred

CS LEHMAN COLLEGE AND GRADUATE CENTER, CITY UNIVERSITY NEW YORK, Bronx, NY, 10468, USA

SO Special Publication - Royal Society of Chemistry (1994), 143(CURRENT TOPICS IN THE CHEMISTRY OF BORON), 185-8
CODEN: SROCDO; ISSN: 0260-6291

DT Journal

LA English

IT 2622-89-1, Diphenylborinic acid

RL: BAC (Biological activity or effector, except adverse); BPR (Biological process); BSU (Biological study, unclassified); CAT (Catalyst use); BIOL (Biological study); PROC (Process); USES (Uses)

(boronic acids as enzyme models and catalysis of oxime formation)

RN 2622-89-1 CAPLUS

CN Borinic acid, diphenyl- (6CI, 7CI, 8CI, 9CI) (CA INDEX NAME)

Patel

9/24/2003>

AB The reactions of several boronic acid compds. and their enzyme-like properties are described.

L4 ANSWER 136 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

AN 1994:523787 CAPLUS

DN 121:123787

TI RuHX(CO)(PR3)2: Can .nu.CO Be a Probe for the Nature of the Ru-X Bond?

AU Poulton, Jason T.; Sigalas, Michael P.; Folting, Kirsten; Streib, William E.; Eisenstein, Odile; Caulton, Kenneth G.

CS Molecular Structure Center, Indiana University, Bloomington, IN, 47405, USA

SO Inorganic Chemistry (1994), 33(7), 1476-85 CODEN: INOCAJ; ISSN: 0020-1669

DT Journal

LA English

IT 156958-65-5

RL: RCT (Reactant); RACT (Reactant or reagent) (reaction of, with ruthenium carbonyl hydrido phosphine complex)

RN 156958-65-5 CAPLUS

CN Borinic acid, bis(2,4,6-trimethylphenyl)-, lithium salt (9CI) (CA INDEX NAME)

● Li

AB The relative electron-donating ability of X in the new 5-coordinate RuHX(CO)(P(CMe3)2Me)2 (X = I, Br, Cl, F, OPh, OH, OCH2CF3, OEt, OCPh3, OB(Mesityl)2, OSiR3, NHPh, SPh, C2Ph) was evaluated based on the CO stretching frequency. In all cases, the CO frequency is lower than that of the free CO and the redn. increases in the order I < Br < Cl < F <alkoxide, with the ethoxide inducing the largest shift. NHPh behaves as OPh and SPh appears at a higher frequency than OPh. Similar measurements were conducted on the 6-coordinate pyridine adducts and a similar ranking of CO frequencies is obtained, but all frequencies are shifted lower. Hydride is the weakest of all donors. There can be no conventional (2-center) Ru-X .pi.-bonding in these pyridine adducts because the Ru(d)/X(p.pi.) interactions are 2-orbital/four-electron ones and thus are net destabilizing. However, the CO .pi.* orbitals interact to stabilize the Ru-X .pi.* orbital, thereby retaining some degree of net Ru-X .pi.. bonding. The structure of RuH(OSiPh3)(CO)(P(CMe3)2Me)2 is square pyramidal with H at the apical site of the pyramid and the siloxy group

9/24/2003>

trans to CO. EHT and core-potential ab initio calcns. (full optimization at the HF and partial optimization at the MP2 level) were performed to det. the structure of RuHX(CO)(PH3)2(X = F, Cl, OH, OMe, OSiH3 with dorbitals on the Si atom). The square pyramid is preferred for all X. This structure permits optical push-pull effects between the .pi.-donating X group and the .pi.* of CO. The CO frequency was calcd. at the MP2 level, and the ranking is identical to the exptl. ones. .pi. Effects are larger for alkoxy than for halide, but the variation in .pi.-effects alone is not sufficient to account for the ranking of the CO frequencies down a column of the periodic table; the .sigma. effect is also involved. In particular, strongly ionic Ru-X bonds lead to lower CO frequency. This last effect causes large changes in the 19F chem. shift for RuH(F)(CO)(P(CMe3)2Me)2 species upon adding a 6th ligand to Ru. It also causes strong H bonding of fluoride to added alc. In spite of Ru/X multiple bonding in the 5-coordinate species, they are Lewis acidic toward amines and phosphines. Crystal data for RuH(OSiPh3)(CO)(P(CMe3)2Me)2 at -160.degree.: a 11.263(1), b 31.713(4), c 22.311(3) .ANG., and .beta. 100.07(0).degree. with Z = 4 in space group P21/n.

L4 ANSWER 137 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

AN 1994:509594 CAPLUS

DN 121:109594

TI NMR studies of 1,1-diphenylboroxazolidone derivatives of .alpha.-amino acids

AU Farfan, Norberto; Silva, David; Santillan, Rosa

CS Cent. Invest. Estud. Avanzados, Inst. Politec. Nac., Mexico City, 07000,

SO Heteroatom Chemistry (1993), 4(6), 533-6

CODEN: HETCE8; ISSN: 1042-7163

DT Journal

LA English

IT 2622-89-1, Diphenylborinic acid

RL: RCT (Reactant); RACT (Reactant or reagent)

(cyclocondensation of, with amino acids, boroxazolidones from)

RN 2622-89-1 CAPLUS

CN Borinic acid, diphenyl- (6CI, 7CI, 8CI, 9CI) (CA INDEX NAME)

GI

AB A series of twelve 1,1-diphenylboroxazolidones I (R = amino acid side chain), prepd. from .alpha.-amino acids H2NCHRCO2H and diphenylborinic acid were studied using one- and, in some cases, two-dimensional (HETCOR)

 ${\tt NMR}$ techniques. Interpretation of these spectra led to definitive assignment of all carbon, hydrogen, and boron resonances.

L4 ANSWER 138 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

AN 1994:459934 CAPLUS

DN 121:59934

TI Aromatic boronic acids as wood preservatives, including solid state NMR studies

AU Meinhold, R. H.

CS N.Z.

SO Ind. Res. Ltd. Rep. (1993), 89, 41 pp. CODEN: IRLRED

DT Report

LA English

IT 71173-48-3

RL: USES (Uses)

(wood preservative activity of)

RN 71173-48-3 CAPLUS

CN Borinic acid, diphenyl-, compd. with 2-aminoethanol (1:1) (9CI) (CA INDEX NAME)

CM 1

CRN 2622-89-1 CMF C12 H11 B O

Ph | Ph-B-OH

CM 2

CRN 141-43-5 CMF C2 H7 N O

 $H_2N-CH_2-CH_2-OH$

AB Styrylboronic acid (I) was obtained by Grignard reaction of p-bromostyrene and B(OBu)3 and its NMR spectra and crystal structure indicated the presence of H bonding. Solid-state photopolymn. of I provided the polymer. I when evaluated (along with other boronic acids) as a pinewood preservative polymd. in situ. The most effective of the 8 compds. tested were PhB(OH)2 and p-BrC6H4B(OH)2.

L4 ANSWER 139 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

AN 1994:436424 CAPLUS

DN 121:36424

TI Tris(pentafluorophenyl)borane complexes and catalysts derived therefrom

IN Siedle, Allen R.; Lamanna, William M.

PA Minnesota Mining and Mfg. Co., USA

SO PCT Int. Appl., 37 pp.

. CODEN: PIXXD2

DT Patent

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LΑ
     English
FAN.CNT 1
      PATENT NO.
                         KIND DATE
                                                  APPLICATION NO.
                                                                      DATE
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PΙ
     WO 9321238
                          ·A2
                                19931028
                                                  WO 1993-US2099
                                                                      19930308
     WO 9321238
                          A3
                                 19940120
          W: AT, AU, BB, BG, BR, CA, CH, CZ, DE, DK, ES, FI, GB, HU, JP, KP, KR, LK, LU, MG, MN, MW, NL, NO, NZ, PL, PT, RO, RU, SD, SE, SK, UA RW: AT, BE, CH, DE, DK, ES, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, BF, BJ, CF, CG, CI, CM, GA, GN, ML, MR, SN, TD, TG
                                                  US 1992-868041
                                                                      19920414
     US 5296433
                          Α
                                19940322
                                                  US 1992-868041
                                                                      19920414
                                                  AU 1993-37971
     AU 9337971
                          A1
                                 19931118
                                                                      19930308
                                                  US 1992-868041
                                                                      19920414
                                                  WO 1993-US2099
                                                                      19930308
     EP 636151
                          Α1
                                19950201
                                                  EP 1993-907329
                                                                      19930308
     EP 636151
                          В1
                                 19971015
          R: BE, CH, DE, FR, GB, IT, LI, NL
                                                  US 1992-868041
                                                                      19920414
                                                  WO 1993-US2099
                                                                      19930308
     JP 07508298
                           T2
                                 19950914
                                                  JP 1993-518312
                                                                      19930308
                                                  US 1992-868041
                                                                      19920414
                                                  WO 1993-US2099
                                                                      19930308
     US 5416177
                           Α
                                 19950516
                                                  US 1994-282820
                                                                       19940729
                                                  US 1992-868041
                                                                       19920414
                                                  US 1993-99197
                                                                      19930729
OS
     MARPAT 121:36424
ΙT
     147892-18-0P 155962-39-3P 155962-41-7P
     155962-42-8P 155962-43-9P 155962-44-0P
     155962-45-1P 155962-47-3P
     RL: PREP (Preparation)
         (prepn. of, as catalysts for polymn. of olefins)
RN
     147892-18-0 CAPLUS
CN
     Borate(1-), hydroxytris(pentafluorophenyl)-, (T-4)-, hydrogen, compd. with
     N, N-diethylethanamine (1:1) (9CI) (CA INDEX NAME)
     CM
           1
     CRN
           147892-17-9
     CMF
           C18 H B F15 O . H
     CCI
           CCS
```

$$F \longrightarrow F \longrightarrow F \longrightarrow F \longrightarrow F$$

● H+

CM 2

CRN 121-44-8 CMF C6 H15 N

RN 155962-39-3 CAPLUS

CN Boron, (1-octadecanol)tris(pentafluorophenyl)-, (T-4)- (9CI) (CA INDEX NAME)

RN 155962-41-7 CAPLUS

CN Boron, (2-butoxyethanol-O1)tris(pentafluorophenyl)-, (T-4)- (9CI) (CA INDEX NAME)

RN 155962-42-8 CAPLUS

CN Boron, (cyclohexanone oxime-O)tris(pentafluorophenyl)-, (T-4)- (9CI) (CA INDEX NAME)

RN 155962-43-9 CAPLUS

CN Boron, [1,1,1,5,5,5-hexamethyl-3-[(trimethylsilyl)oxy]-3-trisiloxanol]tris(pentafluorophenyl)-, (T-4)- (9CI) (CA INDEX NAME)

$$F \longrightarrow F \longrightarrow F \longrightarrow F \longrightarrow F$$

RN 155962-44-0 CAPLUS
CN Boron, aquatris(pentafluorophenyl)-, dihydrate, (T-4)- (9CI) (CA INDEX NAME)

●2 H₂O

RN 155962-45-1 CAPLUS CN Boron, aquatris(pentafluorophenyl)-, (T-4)- (9CI) (CA INDEX NAME)

$$F \xrightarrow{C^{-}} B \xrightarrow{3+} C \xrightarrow{F} F$$

$$F \xrightarrow{F} F$$

RN 155962-47-3 CAPLUS

CN Boron, (methanol)tris(pentafluorophenyl)-, (T-4)-, compd. with methanol (1:1) (9CI) (CA INDEX NAME)

CM 1

CRN 155962-46-2 CMF C19 H4 B F15 O CCI CCS

CM 2

CRN 67-56-1 CMF C H4 O

нзс-он

AB Catalysts for polymn. of olefins comprise complexes of tris(pentafluorophenyl)borane with H2O, alcs., etc. and group IVB organometallic compds. The catalysts are sol. in olefins. 1-Hexene was polymd. using (C6F5)3B.cntdot.H2O and (C5H5)2ZrMe2 catalysts to give a polymer with no.-av. mol. wt. 400.

L4 ANSWER 140 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

AN 1994:270807 CAPLUS

DN 120:270807

```
Derivatives of benzeneborinic acid, preparation thereof and use thereof as
ΤI
     synthetic intermediates
ΙN
```

Chekroun, Isaac; Ruiz-Montes, Jose; Bedoya-Zurita, Manuel; Rossey, Guy

PA Synthelabo S. A., Fr.

SO U.S., 3 pp. CODEN: USXXAM

DTPatent

LΑ English

FAN CNT 1

FAN.	CNT 1				
	PATENT NO.	·KIND	DATE	APPLICATION NO.	DATE
ΡI	US 5278312	Α	19940111		19921029
				FR 1992-12166	19921012
	FR 2696746	A1	19940415	FR 1992-12166	19921012
	FR 2696746	B1	19941118		
	EP 593332	A1	19940420	EP 1993-402424	19931004
	EP 593332	B1	19980114		
	R: AT, BE,	CH, DE	, DK, ES, FR	, GB, GR, IE, IT, LI	, LU, MC, NL, PT, SE
				FR 1992-12166	
	AT 162192	E	19980115	AT 1993-402424	19931004
				FR 1992-12166	19921012
	FI 9304468	Α	19940413	FI 1993-4468	• •
				FR 1992-12166	
	IL 107242	A1	19981030		19931011
				FR 1992-12166	
	CA 2108231	· AA	19940413	CA 1993-2108231	
					19921012
	JP 06192240	A2	19940712		19931012
	01 00132210	212	10010112	FR 1992-12166	
	US 5382672	A	19950117	US 1993-155170	
		A	1990011/		
			•	FR 1992-12166	19921012

OS MARPAT 120:270807

RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)

US 1992-967908

19921029

(prepn. and coupling reaction of, with (bromophenyl)pyrimidinone

ΙT 154466-55-4P

RN154466-55-4 CAPLUS

Borinic acid, bis[2-[2-(1,1-dimethylethyl)-2H-tetrazol-5-yl]phenyl]- (9CI) CN (CA INDEX NAME)

GI

AB A process for the prepn. of derivs. of benzeneborinic acid corresponding to the formula I in which R1, R2 and R3 represent, each independently of the others, either a (C1-C2)alkyl group or an aryl group, the group -CR1R2R3 being in position 1 or 2 of the tetrazole ring and method of use as synthetic intermediates.

9/24/2003>

L4 ANSWER 141 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

AN 1994:264953 CAPLUS

DN 120:264953

TI Covalent protein crosslinks: general detection, quantitation, and characterization via modification with diphenylborinic acid

Ι

AU Graham, Lila; Gallop, Paul M.

CS Dep. Orthop. Surg., Child. Hosp., Boston, MA, 02115, USA

SO Analytical Biochemistry (1994), 217(2), 298-305

CODEN: ANBCA2; ISSN: 0003-2697

DT Journal

LA English

IT 2622-89-1, Diphenylborinic acid

RL: ANST (Analytical study)

(protein covalent crosslinks anal. with)

Page 226

RN 2622-89-1 CAPLUS

CN Borinic acid, diphenyl- (6CI, 7CI, 8CI, 9CI) (CA INDEX NAME)

Ph | | Ph-- B-- OH

AB Progressive crosslinking of proteins appears to be a general phenomenon in aging cells and tissues. Crosslinked proteins can form insol. aggregates which become increasingly resistant to proteolysis as more crosslinks form. However, most evidence for progressive crosslinking with age is indirect, and little is known about the chem. mechanisms involved. The authors have therefore developed a method for detection and isolation of any type of stable covalent crosslink from protein hydrolyzates which requires no prior knowledge of the mol. structure of whatever crosslink(s) may be present. It utilizes the specificity of the diphenylborinic acid reagent for .alpha.-amino acid groups and the chromatog. properties and UV absorbance of the crosslink derivs. The method is demonstrated using 8 different crosslinks from collagen and fibrin, and a general procedure is given for detection of any type of crosslink in a protein hydrolyzate.

L4 ANSWER 142 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

AN 1994:191761 CAPLUS

DN 120:191761

TI Barriers to rotation about the B-X bonds of coordinatively unsaturated borates and thioborates R2BXR' (X=0, S) are not measures of the relative strengths of their B:0 and B:S .pi. bonds

AU Ashby, Michael T.; Sheshtawy, Nader A.

CS Dep. Chem. Biochem., Univ. Oklahoma, Norman, OK, 73019, USA

SO Organometallics (1994), 13(1), 236-43 CODEN: ORGND7; ISSN: 0276-7333

DT Journal

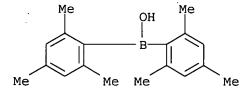
LA English

OS CASREACT 120:191761

IT 20631-84-9P

RN 20631-84-9 CAPLUS

CN Borinic acid, bis(2,4,6-trimethylphenyl) - (9CI) (CA INDEX NAME)



AB The mol. structures of (2,4,6-Me3C6H2)2BXCH3 (1; X = 0, S) have been detd. by single-crystal x-ray crystallog. The boron atoms adopt approx. trigonal planar geometries, and the XC moieties lie in the C2BX planes, an orientation about the B-X bond that maximizes Bp.pi.-Xp.pi. bonding. The mesitylene rings are rotated .apprx.60.degree. with respect to the C2BX plane, which prohibits significant Bp.pi.-aryl interaction. Thus, the crystal structures of 1 (X = 0, S) offer benchmarks for comparing discrete

10085368.2

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Bp.pi.-Xp.pi. bonds. A comparison of the B and X effective radii (calcd. by assuming the B-C and X-C lengths represent single bonds) indicates that the B-O bond is stronger than the B-S bond. Ab initio MO calcns. have been carried out on the model compds. H2BXH (2; X = 0, S). The geometries of 2 have been optimized at the SCF level for various rotational orientations about the B-X bonds. The ground-state geometries of 2 are analogous to those obsd. exptl., with the X-H bonds lying in the trigonal planes of the boron atoms. Mirroring the dynamic behavior obsd. exptl., the energy barrier found for rotation about the B-X bond of 2 (X = S) is larger than that for 2(X = 0). Mulliken population anal. suggests, with respect to the BH2 .pi.-acceptor moiety, that the OH and SH groups are comparable .pi. donors in the ground-state geometry (H-B-X-H=0,180.degree.), but the OH group is a much better .pi. donor than the SH group in the transition-state geometry (H-B-X-H = 90.degree.). Thus the trend in the barriers to rotation is attributed to a greater stabilization of the transition state by oxygen and not a stronger Bp.pi.-Sp.pi. bond in the ground state. Accordingly, rotational barriers about the B-X bonds of R2BOR' and R2BSR' complexes are not measures of their relative B-X .pi.-bond strengths.

L4 ANSWER 143 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

AN 1994:93915 CAPLUS

DN 120:93915

TI Triple bonds between tungsten atoms with ancillary dimesitylboroalkoxide ligands. Preparations, properties and structures of W2(NMe2)4[OB(Mes)2]2 and W2(OBut)4[OB(Mes)2]2

AU Chisholm, Malcolm H.; Folting, Kirsten; Haubrich, Scott T.; Martin, James D.

CS Department of Chemistry and Molecular Structure Center, Indiana University, Bloomington, IN, 47405, USA

SO Inorganica Chimica Acta (1993), 213(1-2), 17-24 CODEN: ICHAA3; ISSN: 0020-1693

DT Journal

LA English

RN

IT 20631-84-9

RL: RCT (Reactant); RACT (Reactant or reagent) (reaction of, with tungsten amido or butoxo dinuclear complexes) 20631-84-9 CAPLUS

CN Borinic acid, bis(2,4,6-trimethylphenyl) - (9CI) (CA INDEX NAME)

AB From the reaction between W2(NMe2)6 and dimesitylborinic acid, (Mes)2BOH (2 equiv), in toluene, the golden-yellow cryst. compd.
W2(NMe2)4[OB(Mes)2]2 (1) was isolated and characterized (elemental anal., 1H, 13C{1H}, 11B NMR spectroscopy, IR spectroscopy and a single crystal x-ray diffraction study). At -160.degree., a 39.379(7), b 13.649(2), c 21.918(4) .ANG., .beta. 123.12(1).degree., Z = 8 and space group C2/c. W2(O(CMe3))4[OB(Mes)2]2 (2) was similarly characterized as a dark red cryst. compd. obtained from the reaction between W2(O(CMe3))6 and (Mes)2BOH (2 equiv) in toluene. At -159.degree., a 17.164(2), b

19.773(2), c 18.490(2) .ANG., .beta. 102.91(1).degree., Z = 4 and space group C2. In both compds. there are unsupported W.tplbond.W bonds of distance 2.3068(13) and 2.3521(15) .ANG. for compd. 1 and 2, resp. In 1 there is a central ethane like W2N4O2 core with the gauche conformation, W-N = 1.94(2) (av.) and W-O = 1.93(1) .ANG.. The coordination geometry at N is trigonal planar and the NC2 units are aligned along the W-W axis as found for related compds. In compd. 2 the W2O4O2 skeleton is eclipsed and most surprisingly the W-O' distances to the OB(Mes)2 ligands are shorter 1.81(1) .ANG. than the W-O distances 1.94(1) and 1.90(1) .ANG. to the alkoxide ligands. In both 1 and 2 the coordination geometry at B is trigonal planar and the O-B distances fall in the range 1.37(3)-1.41(3) .ANG., and are statistically equiv. to that of the free borinic acid. Compd. 2 has crystallog. imposed C2 symmetry and the OB(Mes)2 groups are syn. These are the 1st structurally characterized boroalkoxides coordinated to the (W.tplbond.W)6+ moiety and comparisons of W-O .pi. bonding are made with respect to related OR and OSiR3 compds.

L4 ANSWER 144 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

AN 1994:76952 CAPLUS

DN 120:76952

TI Hindered organoboron groups in organic chemistry. 23. The interactions of dimesitylboron-stabilized carbanions with aromatic ketones and aldehydes to give alkenes

AU Pelter, Andrew; Buss, Dieter; Colclough, Eamon; Singaram, Bakthan

CS Dep. Chem., Univ. Coll. Swansea, Singleton Park/Swansea, SA2 8PP, UK

SO Tetrahedron (1993), 49(32), 7077-103 . CODEN: TETRAB; ISSN: 0040-4020

DT Journal

LA English

OS CASREACT 120:76952

IT 20631-84-9P

RN 20631-84-9 CAPLUS

CN Borinic acid, bis(2,4,6-trimethylphenyl) - (9CI) (CA INDEX NAME)

AB Dimesitylboron-stabilized carbanions react with diaryl ketones to give the corresponding alkenes in mild conditions with good yields. Reactions with arom. aldehydes are more complex, but in all cases E-alkenes are available in good yields by trapping the intermediates with chlorotrimethylsilane followed by treatment with aq. HF/CH3CN. Treatment of the same intermediates with trifluoroacetic anhydride gives mainly the Z-alkenes. The design and mechanisms of these important processes are considered.

L4 ANSWER 145 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

AN 1993:495588 CAPLUS

DN 119:95588

TI Synthesis of di[3,5-di(2,4-dihydroxyphenylazo)phenyl]boric acid

AU Lu, Kui

Page 229

CS Dep. Basic Courses, Zhengzhou Grain Coll., Zhengzhou, 450052, Peop. Rep. China

SO Huaxue Shiji (1993), 15(2), 115-16 CODEN: HUSHDR; ISSN: 0258-3283

DT Journal

LA Chinese

OS CASREACT 119:95588

IT 149054-30-8P

RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)

(prepn. and redn. of)

RN . 149054-30-8 CAPLUS

CN Borinic acid, bis(3,5-dinitrophenyl) - (9CI) (CA INDEX NAME)

IT 149054-32-0P

RL: PRP (Properties); SPN (Synthetic preparation); PREP (Preparation) (prepn. and spectra of)

RN 149054-32-0 CAPLUS

CN Borinic acid, bis[3,5-bis[(2,4-dihydroxyphenyl)azo]phenyl]- (9CI) (CA INDEX NAME)

IT 149054-31-9P

RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)

(prepn., diazotization, and coupling reaction of)

RN 149054-31-9 CAPLUS

CN Borinic acid, bis(3,5-diaminophenyl) - (9CI) (CA INDEX NAME)

Patel 9/24/2003>

GΙ

HO
$$N=N$$
 $N=N$ N

AB The title compd. (I) was prepd. starting for Ph2BOCH2CH2NH2 by nitration, redn., diazotization, and coupling with resorcinol.

L4 ANSWER 146 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

AN 1993:472216 CAPLUS

DN · 119:72216

TI Hindered organoboron groups in organic chemistry. 20. Alkylations and acylations of dimesitylboron stabilized carbanions

AU Pelter, Andrew; Warren, Lorraine; Wilson, John W.

CS Dep. Chem., Univ. Swansea, Swansea, SA2 8PP, UK

SO Tetrahedron (1993), 49(14), 2988-3006 CODEN: TETRAB; ISSN: 0040-4020

DT Journal

LA English

OS CASREACT 119:72216

IT 20631-84-9P

RL: FORM (Formation, nonpreparative); PREP (Preparation) (formation of, by oxidn. of mesitylboron-substituted carbanion)

RN 20631-84-9 CAPLUS

CN Borinic acid, bis(2,4,6-trimethylphenyl) - (9CI) (CA INDEX NAME)

Patel

Ι

AB The alkylation of carbanions derived from alkyldimesitylboranes is an efficient process leading to prim-RBMes2, sec-RBMes2 and tert-RBMes2, all of which can be derived from MeBMes2. Subsequent oxidn. gives a general synthesis for prim-ROH. Secondary alkanols are also produced on oxidn., but the release of tert-alkanols was not accomplished in an efficient fashion. Studies are given of the acylation of dimesitylboron-stabilized carbanions.

L4 ANSWER 147 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

AN 1993:460231 CAPLUS

DN 119:60231

TI Structure of hydroxo(methyl)bis(.eta.5-pentamethylcyclopentadienyl)tantalu m(V) hydroxotris(pentafluorophenyl)borate

AU 'Schaefer, William P.; Quan, Roger W.; Bercaw, John E.

CS Div. Chem. Chem. Eng., California Inst. Technol., Pasadena, CA, 91125, USA

SO Acta Crystallographica, Section C: Crystal Structure Communications (1993), C49(5), 878-81

CODEN: ACSCEE; ISSN: 0108-2701

DT Journal

LA English

IT 148657-99-2

RL: PRP (Properties)
(crystal structure of)

RN 148657-99-2 CAPLUS

CN Tantalum(1+), hydroxymethylbis[(1,2,3,4,5-.eta.)-1,2,3,4,5-pentamethyl-2,4-cyclopentadien-1-yl]-, (T-4)-hydroxytris(pentafluorophenyl)borate(1-) (9CI) (CA INDEX NAME)

CM 1

CRN 148657-98-1 CMF C18 H B F15 O CCI CCS

$$F \longrightarrow C \longrightarrow B \longrightarrow F \longrightarrow F$$

$$F \longrightarrow F \longrightarrow F$$

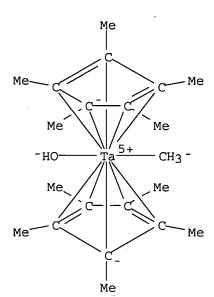
$$F \longrightarrow F$$

$$F \longrightarrow F$$

$$F \longrightarrow F$$

CM 2

CRN 148657-97-0 CMF C21 H34 O Ta CCI CCS



The title compd. is monoclinic, space group P21/n, a 12.217(2), b 16.848(6), c 18.834(3) .ANG., .beta. 100.37(2).degree., Z = 4, dc = 1.75, room temp., R = 0.031 for 3534 reflections. At. coordinates are given. The Ta cation has the expected geometry, with Ta-C and Ta-O distances 2.211(6) and 1.865(5) .ANG., resp. The anion was not characterized previously; its geometry is irregular with tetrahedral angles at B ranging from 103.6(6) to 113.8(6).degree. and systematic angular distortions in the C6F5 rings.

L4 ANSWER 148 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

AN 1993:255062 CAPLUS

DN 118:255062

TI Structure of a zirconoxyborane having a zirconium-fluorine-carbon bridge

AU Siedle, A. R.; Newmark, R. A.; Lamanna, W. M.; Huffman, J. C.

CS 3M Corp. Res. Lab., St. Paul, MN, 55144, USA

SO Organometallics (1993), 12(5), 1491-2 CODEN: ORGND7; ISSN: 0276-7333

DT Journal

LA English

IT 147892-18-0

RL: RCT (Reactant); RACT (Reactant or reagent)
 (reaction of, with dimethylzirconocene)

RN 147892-18-0 CAPLUS

CN Borate(1-), hydroxytris(pentafluorophenyl)-, (T-4)-, hydrogen, compd. with N,N-diethylethanamine (1:1) (9CI) (CA INDEX NAME)

CM 1

CRN 147892-17-9 CMF C18 H B F15 O . H CCI CCS

H+

CM

CRN 121-44-8 CMF C6 H15 N

AΒ Reaction of [Et3NH] [(C6F5)3BOH] with (Me5C5)2ZrMe2 produces methane and (Me5C5)2ZrOB(C6F5)3. The structure reveals a (C6F5)3B-O moiety coordinated to zirconium. One of the C6F5 groups is oriented so that its ortho fluorine forms a Zr-F-C bridge. The 19F NMR spectrum at -88.degree. reveals a static structure in which the shielded Zr-F-C fluorine resonates at -190.3 ppm. As the temp. is raised, all 3 C6F5 rings interconvert at the same rate in a process for which .DELTA.G.thermod. = 10 .+-. 0.5 kcal mol-1.

L4ANSWER 149 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

AN 1993:131359 CAPLUS

DN 118:131359

ΤI Use of hydroxytriphenylborates to treat waste streams

IN Sullivan, Jeffrey M.

PA USA

SO

U.S., 2 pp. CODEN: USXXAM

DT Patent

LΑ English

FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
ΡI	US 5144063	Α	19920901	US 1991-729143	19910712
				US 1991-729143	19910712

ΙT 146142-75-8 146438-27-9

Patel

9/24/2003>

10085368.2

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RL: PROC (Process)

(for cesium removal from wastewaters, by pptn.)

RN 146142-75-8 CAPLUS

CN Borate(1-), hydroxytriphenyl-, cesium, (T-4)- (9CI) (CA INDEX NAME)

• Cs+

RN 146438-27-9 CAPLUS

CN Borate(1-), hydroxytris(methylphenyl)-, cesium (9CI) (CA INDEX NAME)

3 (D1-Me)

• Cs+ .

AB The Cs contained in the waste streams, e.g., from nuclear fission plant effluents or cesium ore digestion, is removed by adding hydroxytriarylborate ions to form a cesium hydroxytriarylborate ppt. (e.g., cesium hydroxytriphenylborate).

L4 ANSWER 150 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

AN 1993:102026 CAPLUS

DN 118:102026

TI Structural studies of organoboron compounds. LV. N,N'-Di-tert-butyl-N,N'-dihydroxypropane-1,3-diamine(O-B)ethoxydiphenylborane and N1,N2-di-tert-butyl-N1,N2-dihydroxy-1-phenylpropane-1,3-diamine(N2-O-B)hydroxydiphenylborane

AU Kliegel, Wolfgang; Lubkowitz, Gottfried; Rettig, Steven J.; Trotter, James

CS Inst. Pharm. Chem., Tech. Univ. Braunschweig, Braunschweig, 3300, Germany SO Canadian Journal of Chemistry (1992), 70(7), 2033-9

Canadian Journal of Chemistry (1992), 70(7), 2033-9 CODEN: CJCHAG; ISSN: 0008-4042

DT Journal

LA English

OS CASREACT 118:102026

IT 146026-50-8P

RL: PRP (Properties); SPN (Synthetic preparation); PREP (Preparation) (prepn. and crystal structure of)

RN 146026-50-8 CAPLUS

CN Borate(1-), [N,N'-bis(1,1-dimethylethyl)-N,N'-dihydroxy-1-phenyl-1,3-propanediaminato-O3]hydroxydiphenyl-, hydrogen, (T-4)- (9CI) (CA INDEX NAME)

● H+

GΙ

The 1,3-propanediamine derivs. N,N'-di-tert-butyl-N,N'-dihydroxy-1,3-propanediamine and N,N'-di-tert-butyl-N,N'-dihydroxy-1-phenyl-1,3-propanediamine were prepd. and reacted with oxybis(diphenylborane) to yield, resp., the cryst. organoboron title compds. I (R = H, Ph). The crystal structure detn. of I (R = H, Ph) showed that the compds. are open-chain hydroxylamine adducts of diphenylborinic acid and Et diphenylborinate, resp. Both of these compds. are stabilized by a system of three intramol. hydrogen bonds (O-H.cntdot..cntdot..cntdot.0, N-H.cntdot..

L4 ANSWER 151 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

AN 1992:426608 CAPLUS

DN 117:26608

TI Through-bond modulation of N .fwdarw. B ring formation shown by NMR and x-ray diffraction studies of borate derivatives of pyridyl alcohols

AU Farfan, Norberto; Castillo, Dolores; Joseph-Nathan, Pedro; Contreras,

Rosalinda; Szentpaly, Laszlo V.

- CS Cent. Invest. Estud. Avanzados, Inst. Politec. Nac., Mexico City, 07000, Mex.
- SO Journal of the Chemical Society, Perkin Transactions 2: Physical Organic Chemistry (1972-1999) (1992), (4), 527-32 CODEN: JCPKBH; ISSN: 0300-9580
- DT Journal
- LA English
- OS CASREACT 117:26608
- IT 2622-89-1, Diphenylborinic acid 141696-41-5
 RL: RCT (Reactant); RACT (Reactant or reagent)
 (cyclocondensation reaction of, with pyridyl alcs.)
- RN 2622-89-1 CAPLUS
- CN Borinic acid, diphenyl- (6CI, 7CI, 8CI, 9CI) (CA INDEX NAME)

RN 141696-41-5 CAPLUS

CN Boron, (2-aminoethanol-N)hydroxydiphenyl-, (T-4)- (9CI) (CA INDEX NAME)

GΙ

AB The prepn. of diphenyl(2-pyridylalkyloxy-0,N)boranes I (X = -, R = H, Me, Ph; X = CH2, R = H), benzene-1,2-diyldioxy(2-pyridylalkyloxy-0,N)boranes II (X = -, CH2) starting from the corresponding pyridyl alcs. are reported. The cyclic structures I (X = -, R = H) and I (X = CH2, R = H) were established by x-ray diffraction studies, the N fwdarw. B bond distances being 1.642 .ANG. and 1.685 .ANG. resp. Complete assignment of the 1H and 13C NMR spectra of compds. was achieved from two dimensional HETCOR data. Addnl. evidence for the strength of the five- and six-membered rings in boron esters was obtained from variable-temp. measurements which show partial ring opening for the six-membered ring compds. at 180.degree.. The observation that five-membered rings are more stable than six-membered ones is attributed to sigma-assistance by

through-bond interactions.

ANSWER 152 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN L4

AN 1992:255804 CAPLUS

DN 116:255804

ΤI Preparation of trisubstituted silylalkyl 1,2,4-triazole and imidazole phenyl borane derivatives

IN Tsang, Tsze H.; Spadafora, Vincent J.

PΑ Chevron Research and Technology Co., USA

SO U.S., 23 pp. CODEN: USXXAM

DT Patent

LΑ English

FAN. CNT 1

L'MIA.	CTA T	_				•	
	PAT	TENT NO.		KIND	DATE	APPLICATION NO.	DATE
ΡI	US	5091377		Α	19920225	US 1990-628806	19901214
	CA	2057193		AA	19920615	CA 1991-2057193	19911206
						US 1990-628806	19901214
	ΕP	490703		· A1	19920617	EP 1991-311629	19911213
		R: AT,	BE,	CH, DE	, DK, ES, F	R, GB, GR, IT, LI, LU	, MC, NL, SE
						US 1990-628806	19901214
	JΡ	05170775	i	A2	19930709	JP 1991-361025	19911216
						US 1990-628806	19901214

OS MARPAT 116:255804

ΙT 12113-07-4

> RL: RCT (Reactant); RACT (Reactant or reagent) (cyclization of)

RN

12113-07-4 CAPLUS
Borate(1-), hydroxytriphenyl-, sodium, (T-4)- (9CI) (CA INDEX NAME) CN

● Na+

GI

$$R^{1}$$
 $R-B^{-}R$
 N^{+}
 R^{5}
 $X-N(CH_{2})_{n}SiR^{2}R^{3}R^{4}$

$$R^{1}$$
 $R-B^{-}R$
 N^{+}
 N^{+}
 R^{5}
 $X-N(CH_{2})_{n}SiR^{2}R^{3}R^{4}$
 I

Title compds. I [R = styryl, (substituted) Ph, (substituted) PhO; R1, R4 = C1-4 alkyl, C3-6 cycloalkyl, C2-6 alkenyl, halo-C3-6-alkenyl; AΒ (substituted) Ph; R2, R3 = (substituted) Ph; R5 = H, C1-4 alkyl; X = HC, N; n = 1, 2], useful as agrochem. fungicides, are prepd. MeMgBr in THF was added under N to 2-aminoethyl diphenyborinate (prepn. given), the mixt. refluxed for 2 h, cooled and treated with 1-[bis(4fluorophenyl)methylsilylmethyl]-1H-1,2,4-triazole to give after work-up I (R = Ph, R1 = R4 = Me, R2 = R3 = 4-FC6H4, R5 = H, X = N; n = 1) (II). II at 200 ppm controlled 100% celery late blight, bean powdery mildew, and bean rust.

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ANSWER 153 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN
L4
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AN 1991:631834 CAPLUS

DN 115:231834

Preparation of haloterphenyls as liquid crystal components ΤI

Poetsch, Eike; Meyer, Volker; Bartmann, Ekkehard; Hittich, Reinhard; INRieger, Bernhard; Reiffenrath, Volker; Coates, David; Greenfield, Simon

PA Merck Patent G.m.b.H., Germany

SO Eur. Pat. Appl., 31 pp.

CODEN: EPXXDW

DT Patent

German LΑ

FAN.	CNT 1			•
	PATENT NO.	KIND	DATE	APPLICATION NO. DATE
ΡI	EP 439089	A1	19910731	EP 1991-100675 19910121
	EP 439089	B1	19950712	•
	R: DE, GB			
				DE 1990-4002145 19900125
				DE 1990-4004098 19900210
	•			DE 1990-4004649 19900215
				DE 1990-4007040 19900307
	DE 4101543	A1	19910801	DE 1991-4101543 19910119
				DE 1990-4002145 19900125
				DE 1990-4004098 19900210
				DE 1990-4004649 19900215
				DE 1990-4007040 19900307

OS MARPAT 115:231834

ΙT 137069-61-5

> RL: RCT (Reactant); RACT (Reactant or reagent) (reaction of, in prepn. of liq. crystal compd.)

RN 137069-61-5 CAPLUS

CN Borinic acid, bis(3,4-difluorophenyl) - (9CI) (CA INDEX NAME)

GI

$$\mathsf{RAZ1}_r - \underbrace{ \begin{array}{c} \mathsf{L1} \\ \mathsf{X} \\ \mathsf{Z} \end{array}} \mathsf{X}$$

AB Title compds. (I; A = trans-1,4-cyclohexylenediyl, 1,4-phenylenediyl, bond; R = CnHzn+1; X = F, Cl, CF3, OCF3, OCF5; 1 of L1,L2,Y,Z = F and the others = H or F; Z1 = C2H4; n = 1-7; r = 0,1) were prepd. Thus, 4-pentyl-2-fluoro-4'-bromobiphenyl was refluxed 6 h with bis(3,4-difluorophenyl)boric acid in PhMe contg. (Ph3P)4Pd and aq. Na2CO3 to give I (R = pentyl, A = bond, L1 = Y = X = F, L2 = Z = H, r = 0).

Ι

L4 ANSWER 154 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

AN 1991:578852 CAPLUS

DN 115:178852

TI Method and apparatus for assay of biogenic amines by HPLC and electrochemical detection

IN Damjanovic, Dragana

PA Can.

SO U.S., 55 pp. CODEN: USXXAM

DT Patent

LA English

FAN.CNT 1

IT 71173-48-3

RL: ANST (Analytical study)

(in biogenic amine extn. for anal. by HPLC with electrochem. detection)

RN 71173-48-3 CAPLUS

CN Borinic acid, diphenyl-, compd. with 2-aminoethanol (1:1) (9CI) (CA INDEX NAME)

CM 1

CRN 2622-89-1 CMF C12 H11 B O Ph | Ph— B— OH

CM 2

CRN 141-43-5 CMF C2 H7 N O

 $H_2N-CH_2-CH_2-OH$

AB A method for assaying biogenic amines, including catecholamines and other small mol. wt. compds., uses a boric acid extn. method followed by HPLC sepn. in conjunction with electrochem. detection. The method utilizes high-purity chem. and liq. components, a microparticulate silica-bonded Ph stationary phase in the chromatog. column, and special cleaning and maintenance measures for the various components of the assaying app., which result in reduced baseline noise and allow the electrochem. cell to be operated at a sensitivity of .ltoreq.1 nA full-scale deflection on a continuous basis.

L4 ANSWER 155 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

AN 1991:440677 CAPLUS

DN 115:40677

TI Synthesis and spectroscopic and x-ray structural characterization of the first homoleptic transition-metal boryloxides [Mn(OBTrip2)(.mu.-OBTrip2)]2 and [Fe(OBMes2)(.mu.-OBMes2)]2

AU Chen, Hong; Power, Philip P.; Shoner, Steven C.

CS Dep. Chem., Univ. California, Davis, CA, 95616, USA

SO Inorganic Chemistry (1991), 30(14), 2884-8 CODEN: INOCAJ; ISSN: 0020-1669

DT Journal

LA English

IT 134627-61-5P 134627-63-7P

RL: PRP (Properties); SPN (Synthetic preparation); PREP (Preparation) (prepn. and crystal structure of)

RN 134627-61-5 CAPLUS

CN Borinic acid, bis[2,4,6-tris(1-methylethyl)phenyl]-, manganese(2+) salt, compd. with hexane (2:1) (9CI) (CA INDEX NAME)

CM 1

CRN 134627-60-4

CMF C30 H47 B O . 1/2 Mn

●1/2 Mn(II)

CM 2

CRN 110-54-3 CMF C6 H14

 Me^- (CH₂)₄ $^-$ Me

RN 134627-63-7 CAPLUS

CN Borinic acid, bis(2,4,6-trimethylphenyl)-, iron(2+) salt, compd. with methylbenzene (1:1) (9CI). (CA INDEX NAME)

CM 1

CRN 134627-62-6

CMF C18 H23 B O . 1/2 Fe

●1/2 Fe(II)

CM 2

CRN 108-88-3 CMF C7 H8

AB The reactions of Mn[N(SiMe3)2]2 and Fe[N(SiMe3)2]2 with the sterically crowded boronous acids Trip2BOH and Mes2BOH (Trip = 2,4,6-i-Pr3C6H2, Mes = 2,4,6-Me3C6H2) afford [Mn(OBTrip2)(.mu.-OBTrip2)]2 (I) and [Fe(OBMes2)(.mu.-OBMes2)]2 (II), which are the first two examples of homoleptic transition-metal boryloxides. The x-ray crystal structures of I and II have also been detd. The data show that both are dimeric with 3-coordinate Mn and Fe centers that are bound to 1 terminal boryloxide ligand and to 2 bridging boryloxide ligands. The M-M distances (3.094(5) for Mn and 3.057(5) .ANG. for Fe) are considerably longer than those found in the amide precursors. Surprisingly, the metric features of I and II are very close to those obsd. in the closely related bis(aryloxo) complexes [M(OAr)2]2 (Ar = 2,4,6-tert-Bu3C6H2, M = Mn, Fe). This suggests that the M-O bonding is similar: furthermore, it is mainly ionic and little evidence for a .pi.-contribution to the M-O bond could be obsd. and II have also been characterized by magnetic measurements. Crystallog. data at 130 K: I.C6H14, a 33.898(11), b 16.985(5), c 30.861(11) .ANG., .beta. 134.65(2).degree., Z = 4, R = 0.088, space group C2/c; II.2C6H5CH3, a 15.004(5), b 14.957(4), c 16.915(6) .ANG., .beta. 93.86(3).degree., Z = 2, R = 0.074, space group P21/n.

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L4 ANSWER 156 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN
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AN 1991:429621 CAPLUS

DN 115:29621

TI Preparation of imidazolyldiphenylborane inner salts as agrochemical fungicides

IN Tsang, Tsze H.; Spadafora, Vincent J.; Pomidor, Patricia

PA Chevron Research and Technology Co., USA

SO U.S., 25 pp. CODEN: USXXAM

DT Patent

LA English

FAN.CNT 1

FAN.	CNT 1			
	PATENT NO.	KIND	DATE	APPLICATION NO. DATE
ΡI	US 4983589	Α	19910108	US 1989-450872 19891214
	US 5051514	Α	19910924	US 1990-587819 19900925
	,			US 1989-450872 19891214
	ZA 9009603	A	19910925	ZA 1990-9603 19901129
				US 1989-450872 19891214
	EP 432987	A2	19910619	EP 1990-313379 19901210
	EP 432987	A3	19911127	
	R: AT, BE,	CH, DE	, DK, ES,	FR, GB, IT, LI, LU, NL, SE
•				US 1989-450872 19891214
	CA 2031980	AA	19910615	CA 1990-2031980 19901211
				US 1989-450872 19891214
	BR 9006317	Α	19910924	BR 1990-6317 19901212
		•		US 1989-450872 19891214
	AU 9067979	A1	19911024	AU 1990-67979 19901212
	AU 635800	B2	19930401	
				US 1989-450872 19891214

OS MARPAT 115:29621

IT 12113-07-4

RL: RCT (Reactant); RACT (Reactant or reagent) (reaction of, in prepri. of agrochem. fungicide)

RN 12113-07-4 CAPLUS

CN Borate(1-), hydroxytriphenyl-, sodium, (T-4)- (9CI) (CA INDEX NAME)

Patel 9/24/2003>

Na +

GI

$$\begin{array}{c|c}
X & R & Y \\
 & & & \\
R^2 & & & \\
N & & & \\
R^1 & & \\
R^4 & & & \\
\end{array}$$

Title compds. I [R = (halo)alkyl, (halo)alkenyl, cycloalkyl; R1 = R, cycloalkyl, hydroxyalkyl, alkoxycarbonylalkyl, (substituted) Ph, PhCH2, 1,3-dioxolan-2-ylalkyl, etc.; R2 = H, alkyl, Ph, (substituted) PhCH2; R3 = H, alkyl; R4 = H, alkyl, Ph, phenylalkyl; X, Y = H, F, Cl, alkoxy, alkylthio, (halo)alkyl], were prepd. Thus, 2-aminoethyl diphenylborinate in THF (prepn. given) was treated with vinylmagnesium bromide and then with 1-vinylimidazole to give title compd. II. II at 2.0 ppm gave 100% control of Erysiphe polygoni on bean seedlings.

L4 ANSWER 157 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

AN 1991:408258 CAPLUS

DN 115:8258

TI Cross-coupling of organometallic compounds with halides or perfluoroalkylsulfonates in the presence of a metallic palladium catalyst

IN Poetsch, Eike; Meyer, Volker; Stahl, Klaus Peter

PA Merck Patent G.m.b.H., Germany

I

SO Ger., 15 pp.

CODEN: GWXXAW

DT Patent

LA German

FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
ΡI	DE 3930663	C1	19901115	DE 1989-3930663	19890914
	DD 297637	A 5	19920116	DD 1990-343980	19900912

Patel 9/24/2003>

10085368.2

Page 244

DE 1989-3930663 19890914 JP 03123736 A2 19910527 JP 1990-242851 19900914 19990621 B2

JP 2907979

DE 1989-3930663 19890914

OS MARPAT 115:8258

IT 134149-94-3P

RL: SPN (Synthetic.preparation); PREP (Preparation) (prepn. and coupling with bromobenzonitrile)

RN 134149-94-3 CAPLUS

CNBorinic acid, bis(4'-pentyl[1,1'-biphenyl]-4-yl)- (9CI) (CA INDEX NAME)

AB Terphenyls, cyclohexylbiphenyls, phenylbicycl heterocycles were prepd. by the title reaction 4-Me(CH2)4C6H4C6H4MgBr-4 reacted with tri-is product treated with 10% aq. HCl to give 4'acid, 5.4 g of which was coupled with 4-BrC6 of Na2CO3 and a Pd/C catalyst in toluene to 4-cyano-4''-pentyl-p-terphenyl.

ANSWER 158 OF 309 CAPLUS COPYRIGHT 2003 ACL L4

AN 1991:207315 CAPLUS

DN 114:207315

TISynthesis of aromatic nitrogen-containing heterocyclic derivatives of asymmetric diarylborinic acids

ΑU Shan, Zixing; Zhao, Dejie; Yuan, Guozheng; Zhang, Guomin

CS Dep. Chem., Wuhan Univ., Wuhan, Peop. Rep. China

Wuhan Daxue Xuebao, Ziran Kexueban (1990), (3), 67-72 SO CODEN: WTHPDI; ISSN: 0253-9888

DT Journal

LΑ Chinese

OS CASREACT 114:207315:

IT 115105-75-4 127844-15-9 133563-62-9 RL: RCT (Reactant); RACT (Reactant or reagent) (reaction of, with pyridinecarboxylic acid)

RN 115105-75-4 CAPLUS

Borinic acid, (4-chlorophenyl)(4-methylphenyl)- (9CI) (CA INDEX NAME) CN

RN 127844-15-9 CAPLUS

Borinic acid, (4-fluorophenyl)(2-methoxyphenyl)- (9CI) (CA INDEX NAME) CN

RN 133563-62-9 CAPLUS

CN Borinic acid, (3-chlorophenyl)phenyl- (9CI) (CA INDEX NAME)

AB Eleven new asym. diarylborinic acids chelated with arom. N-contg. heterocyclic ligands have been synthesized by the reactions of (m-chlorophenyl)phenylborinic acid, (p-chlorophenyl)(p-methylphenyl)borinic acid and (p-fluorophenyl)(o-methoxyphenyl)borinic acid with 2-pyridinecarboxylic acid, quinaldinic acid and 8-hydroxyquinoline, 8-hydroxyquinaldine or 5,7-dibromo-8-hydroxyquinoline. All complexes were characterized with elemental anal. and IR spectroscopy.

L4 ANSWER 159 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

AN 1991:82456 CAPLUS

DN 114:82456

TI Boronic acid catalyzed hydrolyses of salicylaldehyde imines

AU Rao, Galla; Philipp, Manfred

CS Lehman Coll., CUNY, Bronx, NY, 10468, USA

SO Journal of Organic Chemistry (1991), 56(4), 1505-12 CODEN: JOCEAH; ISSN: 0022-3263

DT Journal

LA English

IT 2622-89-1, Diphenylborinic acid

RL: CAT (Catalyst use); USES (Uses)

(catalyst, for hydrolysis for salicylaldehyde isoluecine amides, kinetics and mechanism of)

RN 2622-89-1 CAPLUS

CN Borinic acid, diphenyl- (6CI, 7CI, 8CI, 9CI) (CA INDEX NAME)

AB The hydrolysis of isoleucine salicylaldehyde imines 2-HOC6H4C:NCH(CHMeEt)COR (R = OH, NH2) is catalyzed by boric acid, substituted arylboronic acids, and diphenylborinic acid. These reactions show satn. kinetics, allowing the detn. of first-order catalytic consts. and dissocn. consts. Dissocn. consts. reflect single-ionization pK values similar to the pK values of the boronic acids. Binding is best on the acid side of the pK. Use of the Broensted and the Hammett relationships shows that the binding consts. are improved by electron-withdrawing

Patel 9/24/2003>

substituents on the catalysts. Catalytic consts. are nearly independent of pH, of Hammett .sigma., and of the pK of the catalyst. The reactions display no solvent deuterium isotope effect.

L4 ANSWER 160 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

AN 1991:24001 CAPLUS

DN 114:24001

TI A fluorine-19 NMR study of the transmissive ability of boron-containing bridged systems with three- and four-coordinated boron atoms

AU Pombrik, S. I.; Peregudov, A. S.; Kravtsov, D. N.

CS Inst. Organoelement Compd., Moscow, USSR

SO Heteroatom Chemistry (1990), 1(4), 327-32 CODEN: HETCE8; ISSN: 1042-7163

DT Journal

LA English

OS CASREACT 114:24001

IT 131006-52-5P

RL: SPN (Synthetic preparation); PREP (Preparation) (prepn. of)

RN 131006-52-5 CAPLUS

CN Boron, hydroxy(pyridine)bis(3,4,5-trichlorophenyl)-, (T-4)- (9CI) (CA INDEX NAME)

$$\begin{array}{c|c} C1 & C1 & C1 \\ \hline C1 & R & C1 \\ \hline C1 & C1 & C1 \\ \hline \end{array}$$

IT 131112-05-5 131112-06-6

RL: RCT (Reactant); RACT (Reactant or reagent)
 (reaction of, with fluorophenol, fluorophenyl ester of boronic acid
 from)

RN 131112-05-5 CAPLUS

CN Borinic acid, bis(3,4-dichlorophenyl) - (9CI) (CA INDEX NAME)

RN 131112-06-6 CAPLUS

CN Borinic acid, bis(3,4,5-trichlorophenyl) - (9CI) (CA INDEX NAME)

AB Two series of model compds., R2BOC6H4F-4 (R = 4-tolyl, Ph, 4-ClC6H4, 3,4-Cl2C6H3, 3,4,5-Cl3C6H2) and R2B(Py)OC6H4F-4, contg. three- and four-coordinated boron atoms, were synthesized from R2BOH and 4-FC6H4OH. It was established by 19F NMR that both systems possess a similar transmissive ability in that intermol. coordination with pyridine does not influence the transmissive properties of the boron-oxygen binuclear bridging group. The solvent polarity does not influence the electron transmission in the boron complexes studied.

L4 ANSWER 161 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

AN 1990:441252 CAPLUS

DN 113:41252

TI Diaryl boron chelates of thioproline

AU Yuan, Guozheng; Huang, Junjie; Zhang, Guomin

CS Dep. Chem., Wuhan Univ., Wuhan, Peop. Rep. China

SO Gaodeng Xuexiao Huaxue Xuebao (1989), 10(9), 881-5

CODEN: KTHPDM; ISSN: 0251-0790

DT Journal

LA Chinese

IT 66117-64-4 73774-45-5 89566-59-6

96484-29-6 115105-75-4 127844-13-7

127844-14-8 127844-15-9

RL: RCT (Reactant); RACT (Reactant or reagent)

(reaction of, with thioproline)

RN 66117-64-4 CAPLUS

CN Borinic acid, bis(4-methylphenyl) - (9CI) (CA INDEX NAME)

RN 73774-45-5 CAPLUS

CN Borinic acid, bis(4-methoxyphenyl) - (9CI) (CA INDEX NAME)

RN 89566-59-6 CAPLUS

CN Borinic acid, bis(4-chlorophenyl) - (9CI) (CA INDEX NAME)

RN 96484-29-6 CAPLUS

CN Borinic acid, bis(4-bromophenyl) - (9CI) (CA INDEX NAME)

RN 115105-75-4 CAPLUS

CN Borinic acid, (4-chlorophenyl) (4-methylphenyl) - (9CI) (CA INDEX NAME)

RN 127844-13-7 CAPLUS

CN Borinic acid, (4-chlorophenyl)(4-methoxyphenyl)- (9CI) (CA INDEX NAME)

RN 127844-14-8 CAPLUS

CN Borinic acid, (4-fluorophenyl)(4-methoxyphenyl)- (9CI) (CA INDEX NAME)

RN 127844-15-9 CAPLUS

CN Borinic acid, (4-fluorophenyl)(2-methoxyphenyl)- (9CI) (CA INDEX NAME)

Patel

9/24/2003>

GI

AB Nine title chelates, e.g., I were prepd. and characterized.

L4 ANSWER 162 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

AN 1990:411792 CAPLUS

DN 113:11792

TI Photochemical degradation rates of tetraphenylborate and diphenylboric acid sensitized by dissolved organic matter in stream water

AU Mills, Gary L.; Schwind, Donna

CS Savannah River Ecol. Lab., Univ. Georgia, Aiken, SC, 29801, USA

SO Environmental Toxicology and Chemistry (1990), 9(5), 569-74 CODEN: ETOCDK; ISSN: 0730-7268

DT Journal

LA English

IT 2622-89-1

RL: POL (Pollutant); OCCU (Occurrence)

(water pollution by, photochem. degrdn. in, of streams)

RN 2622-89-1 CAPLUS

CN Borinic acid, diphenyl- (6CI, 7CI, 8CI, 9CI) (CA INDEX NAME)

Patel

AB The photochem. degrdn. of tetraphenylborate and diphenylboric acid was studied in stream water and humic acid solns. The rates of indirect photolysis of tetraphenylborate and diphenylboric acid were similar and directly proportional to the concn. of stream water dissolved org. matter and humic acid for values <10 mg/L. Direct photolysis of both organoboron compds. was nondetectable during a 2-h irradn. period. Indirect photolysis rates varied only slightly from pH 6.0 to 9.0, but increased markedly below pH 6.0. Removal of dissolved O increased reaction rates by a factor of 2, indicating that the reaction mechanism does not involve singlet O oxygenation.

. 9/24/2003>

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ANSWER 163 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN
L4
AN
     1990:153203 CAPLUS
DN
     112:153203
ΤI
     Surface-facilitated chemical degradation of tetraphenylboron in soil
ΑU
    Mills, Gary L.; Kaplan, Daniel; Schwind, Donna; Adriano, Domy
     Savannah River Ecol. Lab., Univ. Georgia, Aiken, SC, 29801, USA
CS
     Journal of Environmental Quality (1990), 19(1), 135-40
SO
     CODEN: JEVQAA; ISSN: 0047-2425
DT
     Journal
LΑ
     English
ΙT
     2622-89-1
     RL: BIOL (Biological study)
        (as tetraphenylboron degrdn. product, in soil)
RN
     2622-89-1 CAPLUS
CN
    Borinic acid, diphenyl- (6CI, 7CI, 8CI, 9CI) (CA INDEX NAME)
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Ph | Ph— B— OH

AB Lab. studies were conducted to examine the surface-promoted degrdn. of tetraphenylboron (TPB) in soil. The results indicated that TPB degrades rapidly in soil and that the processes involved are predominately abiotic reactions. The initial products of degrdn. of TPB are diphenylborinic acid (DPBA) and biphenyl. A portion of the DPBA produced subsequently degrades to produce boric acid. However, only about 20% of the total B added initially to the soil as TPB was accounted for by the measured species at the end of the expt. Most of the remaining B appears to be present in moderately stable org. compds. Comparison of the degrdn. rates in different soils indicated that the rates in the surface soil were somewhat higher than in the subsoil. The only measured characteristic of these soils that follows a corresponding trend is soil org. matter content. Addnl. expts. indicated that rates of the initial reaction was inversely related to soil moisture content. Obsd. reaction products indicate that the initial step in the degrdn. process is a two-step oxidn. of Lewis acid sites assocd. with soil component surfaces.

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L4
     ANSWER 164 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN
AN
     1990:132533 CAPLUS
     112:132533
DN
ΤI
     Solvent extraction and high performance liquid chromatography with
     electrochemical detection for determination of plasma catecholamines
AU
     Zhang, Lian; Zhao, Weikang
CS
     Dep. Biochem., Shanghai Coll. Tradit. Chin. Med., Shanghai, 200032, Peop.
     Rep. China
SO
     Zhongguo Yaoli Xuebao (1989), 10(6), 572-5
     CODEN: CYLPDN; ISSN: 0253-9756
DT
     Journal
LΑ
     Chinese
IT
     2622-89-1
     RL: BIOL (Biological study)
```

RN 2622-89-1 CAPLUS CN Borinic acid, diphenyl- (6CI, 7CI, 8CI, 9CI) (CA INDEX NAME)

(in catecholamine extn. from blood plasma, for HPLC)

AB A technique for selectively extg. plasma catecholamines prior to quantification by HPLC-electrochem. detection is described. The extn. system was a 2-stage process. The first stage involved complex formation between the diphenylborate and catechol (diol) groups in alk. medium. second stage was a liq.-liq. extn. The complex combined with tetraoctyl-ammonium bromide to form an ion-pair in org. solvent. catecholamines in turn were extd. with acid. This technique provided a very specific extn. procedure which resulted in chromatograms with few interfering compds. and gave abs. recoveries (100-103%) of norepinephrine, epinephrine, and dopamine. Meanwhile, the plasma catecholamines were concd. and the sensitivity of detection was increased. A good linear relationship was found between the concns. and ratio of peak heights of the catecholamines from 0.125-2 ng. The correlation coeff. ranged 0.998-0.999. The relative std. deviations of the intra- and inter-assay were within 3% and 6%, resp. The results show that the procedure is very simple and fast. The method is valuable not only for clin. diagnosis but also for lab. research.

L4 ANSWER 165 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

AN 1990:118888 CAPLUS

DN 112:118888

TI Bora-aromatic systems. Part 10. Aromatic stabilization of the triarylborirene ring system by tricoordinate boron and facile ring-opening with tetracoordinate boron

AU Eisch, John J.; Shafii, Babak; Odom, Jerome D.; Rheingold, Arnold L.

CS Dep. Chem., State Univ. New York, Binghamton, NY, 13901, USA

SO Journal of the American Chemical Society (1990), 112(5), 1847-53 CODEN: JACSAT; ISSN: 0002-7863

DT Journal

LA English

OS CASREACT 112:118888

IT 20631-84-9P

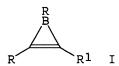
RL: SPN (Synthetic preparation); PREP (Preparation)

(prepn. of)

RN 20631-84-9 CAPLUS

CN Borinic acid, bis(2,4,6-trimethylphenyl)- (9CI) (CA INDEX NAME)

GΙ



AB To remove uncertainties in the apparent C:C and B-C bond lengths of the borirene ring, as previously estd. from an X-ray crystallog. anal. of trimesitylborirene, the unsym. substituted 2-(2,6-dimethylphenyl)-1,3dimesitylborirene I (R = mesityl, R1 = 2,6-Me2C6H3) was prepd. by the photorearrangement of [(2,6-dimethylphenyl)ethynyl]dimesitylborane. With such an unsym. borirene there was no disorder in the identity of the ring atoms, and all three ring atom sepns. could be unambiguously assigned. Compared with the reported C:C bond length in cyclopropene (1.304 .ANG.), the C:C bond in the borirene has been lengthened by 0.08 .ANG., and compared with the reported C-B bond length in triviny.lborane (1.558 .ANG.), ring C-B bonds have been shortened by an av. of 0.10 .ANG.. altered bond lengths are fully consistent with those expected if the two ring .pi.-electrons are extensively delocalized among the three sp2-hybridized ring atoms. Therefore, the borirene ring can truly be considered as a Hueckel arom. nucleus. In order to corroborate that cyclic conjugation led to C-C bond lengthening, the open-chain precursor to the borirene ring, namely dimesityl(mesitylethynyl)borane was also examd. by x-ray crystallog. In this system the C.tplbond.C bond showed no unusual lengthening and hence gave no indication of significant conjugation with the tricoordinate boron. When the tricoordinate boron of the borirene ring becomes tetracoordinate by ligation with an amine, such as pyridine, or a sterically suitable alc. like MeOH or EtOH, the borirene ring is promptly ruptured and, in the presence of a proton source, irreversibly converted into an acyclic borinic ester. Such rupture of the borirene ring by pyridines can be monitored by electronic and multinuclear NMR spectroscopy. These observations corroborate the role of the available 2pz-orbital on sp2-hybridized boron in stabilizing the borirene ring.

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L4 ANSWER 166 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN
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AN 1989:546906 CAPLUS

DN 111:146906

TI A general method for the quantitative determination of catecholamines in body fluids using off-line sample pretreatment and HPLC-ED analysis

AU Bauch, H. J.; Struewer, E.; Kelsch, U.

CS Inst. Arterioskleroseforsch., Univ. Muenster, Muenster, D-4400, Fed. Rep. Ger.

SO Chromatographia (1989), 28(1-2), 78-84 CODEN: CHRGB7; ISSN: 0009-5893

DT Journal

LA English

IT 83075-94-9

RL: BIOL (Biological study)

(in catecholamine-contg. sample prepn. for HPLC)

RN 83075-94-9 CAPLUS

CN Borate(1-), dihydroxydiphenyl-, (T-4)- (9CI) (CA INDEX NAME)

ΑB Reversed phase HPLC with electrochem. detection (HPLC-ED) was used for quant. detn. of adrenaline, noradrenaline, and dopamine in several complex biol. matrixes, including plasma, uremic plasma, and urine. Three different methods of sample prepn. for clin. use . were tested. These were: adsorption of catecholamines on alumina, org. solvent extn. after complex formation with diphenylborate, and adsorption of catecholamines on a cation exchange gel followed by org. solvent extn. of the elute. The selectivity and precision of the 3 methods were evaluated. The org. solvent extn. proved to be more precise and selective than adsorption on alumina (adrenaline: relative std. deviation (rsd) = 3.80 vs. 7.58%; noradrenaline: rsd = 1.7 vs. 4.26%); it also proved suitable for use in the routine quant. detn. of catecholamines in plasma from patients with normal renal function (creatinine <1.2 mg/dL). However when working with uremic plasma or urine, a more selective sample prepn. was required. In this case the adsorption of catecholamines on a cation exchange gel followed by org. solvent extn. of the elute was sufficiently selective and precise and thus allowed a reliable quant. detn. of adrenaline and noradrenaline from rather complex biol. matrixes (adrenaline: rsd = 6.2%; noradrenaline: rsd = 2.8%). This specific method showed that basal plasma catecholamine levels in dialysis patients are comparable to those in patients with normal renal function (adrenaline: 47.7 pg/mL; noradrenaline: 310.3 pg/mL).

L4 ANSWER 167 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

AN 1989:453523 CAPLUS

DN 111:53523

 ${\tt TI}$ Separation and determination of .alpha.-amino acids by boroxazolidone formation

AU Strang, Candace J.; Henson, Edward; Okamoto, Yoshiaki; Paz, Mercedes A.; Gallop, Paul M.

CS Child. Hosp., Harvard Sch. Med., Boston, MA, 02115, USA

SO Analytical Biochemistry (1989), 178(2), 276-86 CODEN: ANBCA2; ISSN: 0003-2697

DT Journal

LA English

IT 2622-89-1

RL: ANST (Analytical study)

(amino acids derivatization by, for reversed-phase HPLC)

RN 2622-89-1 CAPLUS

CN Borinic acid, diphenyl- (6CI, 7CI, 8CI, 9CI) (CA INDEX NAME)

CN Borinic acid, diphenyl- (6CI, 7CI, 8CI, 9CI) (CA INDEX NAME)

AB Reaction of an .alpha.-amino acid (.alpha.-AA) with 1,1-diphenylborinic acid (DPBA) leads to the formation of a kinetically stable adduct at pH 2-5 in which both the .alpha.-amino and the .alpha.-carboxyl groups are bound to B forming a cyclic mixed anhydride termed a boroxazolidone. In this adduct, the >N:B bond is coordinate, involving the free electron pair of N, thereby satisfying the octet rule for the 2nd electron shell of B (Group IIIA). Consequently, the .alpha.-amino function of the boroxazolidone can be primary, secondary, or tertiary, as demonstrated by boroxazolidone formation with glycine, N-methylglycine, and N, N-dimethylqlycine. On reaction with DPBA, the .alpha.-AA moiety of N-terminal .gamma.-qlutamyl peptides is also derivatized as demonstrated by the formation of a glutathione boroxazolidone. The 1,1-diphenylboroxazolidone adducts of .alpha.-AA may be sepd. by reversed-phase (RP)-HPLC (AA-DPBA/RP-HPLC) enabling the derivatization procedure to be used as a precolumn reaction for .alpha.-AA anal. Under the conditions described, DPBA is not stably reactive with the .epsilon.-amino group of lysine. Furthermore, it does not complex with amide bonds of the peptide backbone or to any side chains of the common amino acids. Reaction of an .alpha.-AA mixt. with DPBA, followed by RP-HPLC (AA-DPBA/RP-HPLC) is then a simple method by which to analyze .alpha.-AA in a mixt. with peptides and amines. Precolumn reaction with DPBA may be used to sep. peptides from .alpha.-AA and from those peptides which contain an .alpha.-AA moiety. Unreacted peptides are bound only weakly to the HPLC column and thus are sepd. from reacted .alpha.-amino acids which are retained as 1,1-diphenylboroxazolidones until their selective elution: This method is particularly suited for the anal. of .alpha.-amino acids that are derived from post-translational modification of protein side chains.

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L4 ANSWER 168 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN
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AN 1989:228148 CAPLUS

DN 110:228148

TI Formation of analyzable boron-containing adducts

IN Gallop, Paul M.; Henson, Edward; Flueckiger, Rudolf

PA Children's Medical Center Corp., USA

SO U.S., 11 pp. CODEN: USXXAM

DT Patent

LA English

FAN.CNT 1

1111.011 1										
	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE					
PΙ	US 4713346	Α	19871215	US 1986-825619	19860203					
				US 1986-825619	19860203					

OS CASREACT 110:228148

IT 2622-89-1

RL: RCT (Reactant); RACT (Reactant or reagent) (reaction of, with org. compds. to form analyzable adducts)

RN 2622-89-1 CAPLUS

CN Borinic acid, diphenyl- (6CI, 7CI, 8CI, 9CI) (CA INDEX NAME)

Patel 9/24/2003>

GI

AB A method of forming analyzable adducts in a mixt. of org. compds. comprises contacting the mixt. with a B reagent XB(Y)OH(X, Y =C.ltoreq.12 alkyl, C6-20 aryl) or BZ3 (Z = X). The compds. in the mixt. contain the functionality I (OH is hydroxy or part of carboxyl group; N is part of amino, imino, or arom. heterocyclic group), e.g. protein hydrolyzate, .alpha.-amino acids, 2-carboxypyrazines, .gamma.-glutamyl peptides. Alternatively, the compds. are primary or secondary alcs. or amines or are ketones or aldehydes and are reacted with reagents to form a product contg. the reactive functionality and -C(0)NHN:C1 (C1 = carbonyl C of ketone or aldehyde) before contacting with the B reagent. The anal. is performed by HPLC, TLC, or mass spectroscopy and the adducts are detected by UV absorption. Amino acids were reacted with an equimolar amt. of diphenylborinic acid (DPBA) or the resp. molar excess if other than 1:1 complexes could result, in EtOH: H2O (1:1) at 60-70.degree. for 15 min. Thirteen amino acid: DPBA adducts were mixed together and sepd. by HPLC on .mu.Bondapak C-18.

L4 ANSWER 169 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

AN 1989:108309 CAPLUS

DN 110:108309

TI Automated HPLC catecholamine determination in plasma: clean-up through complex formation with diphenylborinic acid

AU Ni, P.; Guyon, F.; Caude, M.; Rosset, R.

CS Lab. Chim. Anal., Ec. Super. Phys. Chim. Paris, Paris, 75231, Fr.

SO Analusis (1988), 16(9-10), 484-90 CODEN: ANLSCY; ISSN: 0365-4877

DT Journal

LA French

IT 2622-89-1D, Diphenylborinic acid, catecholamine complexes RL: FORM (Formation, nonpreparative)
(formation of, for catecholamine detn. in blood plasma by HPLC)

RN 2622-89-1 CAPLUS

CN Borinic acid, diphenyl- (6CI, 7CI, 8CI, 9CI) (CA INDEX NAME)

AB Detn. of catecholamines (adrenaline, noradrenaline, and dopamine) in plasma can be carried out by extn. and clean-up on precolumn (octadecyl

bonded silica gel), by formation of hydrophobic complexes with diphenylborinate anion in basic medium. The catecholamines were resolved online by ion-pair chromatog. with SDS as counterion and octadecyl bonded silica gel as stationary phase (acetonitrile-phosphate buffer 0.05 M, pH = $2.8,\ 20\text{-}80\ \text{vol./vol.})$. The method can be automated using the AASP system (Advanced Automated Sample Processor). The total anal. time is 20 min. The measured reproducibility is close to 10% for 100 ng/L. The detection limits are 5 ng/L for adrenaline and noradrenaline and 3.5 ng/L for dopamine when using electrochem. detection. The formation of diphenylborinate complexes increases the catecholamine stability. Consequently the cartridge stock duration can reach 12 h at room temp. and 24 h at 5.degree.

L4 ANSWER 170 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

AN 1989:95554 CAPLUS

DN 110:95554

TI Structural studies of organoboron compounds. XXXI. (3-Benzoyl-(+)-camphorato)diphenylboron

AU Cullen, William R.; Rettig, Steven J.; Trotter, James; Wickenheiser, Eugene B.

CS Dep. Chem., Univ. British Columbia, Vancouver, BC, V6T 1Y6, Can.

SO Canadian Journal of Chemistry (1988), 66(8), 2007-13 CODEN: CJCHAG; ISSN: 0008-4042

DT Journal

LA English

OS CASREACT 110:95554

IT 2622-89-1P

RN 2622-89-1 CAPLUS

CN Borinic acid, diphenyl- (6CI, 7CI, 8CI, 9CI) (CA INDEX NAME)

GI

AB Details of the synthesis and phys. properties of the title compd. I are reported along with a new prepn. of 3-benzoyl-(+)-camphor (II). Crystals of (3-benzoyl-(+)-camphorato)diphenylboron are triclinic. The unit-cell contains two crystallog. independent mols., related to one another in the crystal lattice by a pseudo-inversion center, and having the same

configuration but different conformations. The structure anal. shows that the chiral, anionic, 1,3-diketonate ligand derived from II is not sym. delocalized like most related ligands, resulting in significantly different O-B bond lengths. Bond lengths (cor. for libration) include: O-B = 1.514(4), 1.522(4); 1.568(4) and 1.567(4) .ANG.; C-B = 1.594(5)-1.616(5) .ANG..

L4 ANSWER 171 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

AN 1989:19755 CAPLUS

DN 110:19755

TI Fungicidal activity of diphenylboric acid derivatives

AU Molodykh, Zh. V.; Teplyakova, L. V.; Nikonov, G. N.; Erastov, O. A.

CS USSR

SO Fiziologicheski Aktivnye Veshchestva (1988), 20, 68-71 CODEN: FAVUAI; ISSN: 0533-1153

DT Journal

LA Russian

IT 2622-89-1D, glycol esters and glycol ester alkylamine complexes RL: AGR (Agricultural use); BAC (Biological activity or effector, except adverse); BSU (Biological study, unclassified); BIOL (Biological study); USES (Uses)

(fungicidal activity of)

RN 2622-89-1 CAPLUS

CN Borinic acid, diphenyl- (6CI, 7CI, 8CI, 9CI) (CA INDEX NAME)

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Ph
|
Ph— B— OH
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AB Of 4 glycol diphenylborates, HO(CH2)70BPh2 (I) was the most effective. I at 1.7 .times. 10-4M completely suppressed the growth of Aspergillus niger, Fusarium moniliforme, Rhizoctonia solani, and Helminthosporium sativum mycelium. HO(CH2)30BPh2, HO(CH2)60BPh2, and the branched ester, HOCHMeCH2CH2CHMeOBPh2 (II), were less effective. The most effective was the C10H21NH2 salt of II (III). However, III was less effective than I. II depressed lipase, SS, and SH groups of H. sativum more effectively than did III which, however, inhibited dehydrogenase and protein formation more effectively.

L4 ANSWER 172 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

AN 1988:621368 CAPLUS

DN 109:221368

TI Rhenium complexes of tetraaza macrocycles: the synthesis and single-crystal x-ray structure of trans-dioxo(1,4,8,11-tetraazacyclotetradecane)rhenium(V) chloride-(triphenylboron monohydrate)(1/2)

AU Blake, Alexander J.; Greig, John A.; Schroeder, Martin

CS Dep. Chem., Univ. Edinburgh, Edinburgh, EH9 3JJ, UK

SO Journal of the Chemical Society, Dalton Transactions: Inorganic Chemistry (1972-1999) (1988), (10), 2645-7 CODEN: JCDTBI; ISSN: 0300-9246

DT Journal

LA English

IT 117579-38-1P

RL: PRP (Properties); SPN (Synthetic preparation); PREP (Preparation) (prepn. and crystal structure of racemic)

Patel 9/24/2003>

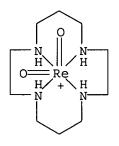
RN 117579-38-1 CAPLUS

CN Rhenium(1+), dioxo(1,4,8,11-tetraazacyclotetradecane-N1,N4,N8,N11)-, [OC-6-13-(1R*,4R*,8S*,11S*)]-, chloride, compd. with (T-4)-aquatriphenylboron (1:2) (9CI) (CA INDEX NAME)

CM 1

CRN 117579-37-0 CMF C10 H24 N4 O2 Re . Cl

CCI CCS



● C1 -

CM 2

CRN 117579-36-9 CMF C18 H17 B O CCI CCS

$$\begin{array}{c|c}
C^{-} & B \\
\hline
C^{-} & C \\
\hline
C^{-} & C
\end{array}$$

Reaction of [ReOCl3(PPh3)2] with 1,4,8,11-tetraazacyclotetradecane (cyclam) in CH2Cl2 affords ReOCl3(cyclam) which hydrolyzes readily to [Re(O)2(cyclam)]+ in soln. trans-[Re(O)2(cyclam)]Cl.2(BPh3.H2O) crystallizes in the monoclinic space group P21/n, a 9.3869(4), b 13.5504(7), c 17.7727(11) .ANG., .beta. 91.918(5).degree., Z = 2, implying that both the macrocyclic complex cation and the chloride counter-ion lie on inversion centers. The single-crystal x-ray structure of the complex shows octahedral ReV with the tetraaza macrocyclic ligand bound in the equatorial plane adopting an RRSS (trans-III) configuration at the coordinated N donors, Re-N(1) 2.128(3), Re-N(11) 2.135(3) .ANG.. The coordination shell is completed by mutually trans dioxo ligands, Re:O 1.756(3) .ANG.. The Cl- counter-ion is not coordinated to the Re center.

Two mols. of the adduct BPh3.H2O are also obsd. in the crystal. Extensive H bonding is obsd. in the crystal between the oxo ligands, H2O mols., Cl counter-ions, and the amine protons of the macrocyclic ligand.

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ANSWER 173 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN
L4
     1988:569394 CAPLUS
AN
DN
     109:169394
TI
     Long-term phytoavailability of soil-applied organo-borates
ΑU
     Adriano, D. C.; Kaplan, D. I.; Burkman, W. G.; Mills, G. L.
CS
     Inst. Ecol., Univ. Georgia, Aiken, SC, 29801, USA
SO
     Journal of Environmental Quality (1988), 17(3), 485-92
     CODEN: JEVQAA; ISSN: 0047-2425
DT
     Journal
     English
LΑ
IT
     2622-89-1
     RL: BIOL (Biological study)
        (plant and soil boron and sorgrass growth response to)
RN
     2622-89-1 CAPLUS
     Borinic acid, diphenyl- (6CI, 7CI, 8CI, 9CI) (CA INDEX NAME)
CN
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AΒ Sodium tetraphenylboron (NaTPB) is expected to be used in large quantities to sep. radiocesium from high-level nuclear wastes. Greenhouse expts. were conducted to det. the long-term effects of NaTPB, diphenylboric acid (DPBA, a major degrdn. byproduct of NaTPB), and boric acid (BA) on the extractability of soil B and plant B nutrition. Sorgrass (Sorghum vulgare sudanense Dub-L-Graze) was planted in sandy and loamy sandy soils (Grossarenic and Typic Paleudults) in 2 sep. 2-yr studies. Initial differences between effects of the B sources on biomass, plant B concn., plant B uptake, and hot-water extractable B disappeared after the 1st harvest, while differences among these parameters due to soil type and application rate remained throughout the expts. Extractable soil and plant B concns. tended to decrease more gradually in the loamy sand than in the sandy soil. Plant toxicity from org. sources was noted only during the 1st harvest while BA had no adverse effects. Both NaTPB and DPBA reduced biomass, the former more than the latter. Initially, plant B concns. were higher in NaTPB than BA treatments. The cumulative percentage of soil-applied B removed after 2 yr by sorgrass remained fairly similar, 20.0 .+-. 1.7% (1 SD) among B sources and application rates. This suggests that a large fraction of B applied to the soil was not taken up by the plant, presumably due to soil fixation. Biphenyl, another major breakdown product of NaTPB, had no effect on sorgrass growth, tissue B concn., or soil B concn.

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L4 ANSWER 174 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN
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- AN 1988:549589 CAPLUS
- DN 109:149589
- TI Structural studies of organoboron compounds. XXVII. (3-Hydroxy-2-methyl-4-pyridinonato)diphenylboron and (3-hydroxy-1,2-dimethyl-4-pyridinonato)diphenylboron
- AU Nelson, William O.; Orvig, Chris; Rettig, Steven J.; Trotter, James
- CS Dep. Chem., Univ. British Columbia, Vancouver, BC, V6T 1Y6, Can.
- SO Canadian Journal of Chemistry (1988), 66(1), 132-8

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10085368.2
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Page 260

CODEN: CJCHAG; ISSN: 0008-4042

DT Journal

LA English

OS CASREACT 109:149589

IT 2622-89-1P, Diphenylborinic acid

RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)

(prepn. and reaction of, with hydroxypyridinone derivs.)

RN 2622-89-1 CAPLUS

CN Borinic acid, diphenyl- (6CI, 7CI, 8CI, 9CI) (CA INDEX NAME)

AB The prepn. (from Ph2BOH and hydroxypyridinone derivs.) and crystal structures of the title compds. (I and II, resp.) are given. Both mols. contain five-membered C2O2B chelate rings, that in II being nearly planar. Structural data indicate weaker overall binding of the ligand O atoms to boron in I than in II.

L4 ANSWER 175 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

AN 1988:454817 CAPLUS

DN 109:54817

TI Boron compounds. (XIX). New organyloxydiarylborane chelates containing the virucide 2-(.alpha.-hydroxyalkyl)benzimidazole as ligands

AU Yuan, Guozheng; Li, Guiying; Zhang, Guomin

CS Dep. Chem., Wuhan Univ., Guomin, Peop. Rep. China

SO Gaodeng Xuexiao Huaxue Xuebao (1987), 8(5), 398-402 CODEN: KTHPDM; ISSN: 0251-0790

DT Journal

LA Chinese

IT 2622-89-1P 66117-64-4P 89566-59-6P

115105-75-4P

RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)

(prepn. and chelation of, with hydroxymethylbenzimidazole)

RN 2622-89-1 CAPLUS

CN Borinic acid, diphenyl- (6CI, 7CI, 8CI, 9CI) (CA INDEX NAME)

RN 66117-64-4 CAPLUS

CN Borinic acid, bis(4-methylphenyl) - (9CI) (CA INDEX NAME)

RN 89566-59-6 CAPLUS

CN Borinic acid, bis(4-chlorophenyl) - (9CI) (CA INDEX NAME)

RN 115105-75-4 CAPLUS

CN Borinic acid, (4-chlorophenyl) (4-methylphenyl) - (9CI) (CA INDEX NAME)

GI

AB Title compds. I (R, R1 = H, Me, C1; R2 = H, Ph) were prepd. through the reaction of 2-(.alpha.-hydroxymethyl)benzimidazole or 2-(.alpha.-hydroxybenzyl)-benzimidazole with diarylborinic acid. These compds. were identified by elemental anal., MS, IR and UV spectroscopies.

L4 ANSWER 176 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

AN 1988:435183 CAPLUS

DN 109:35183

TI Glycolic esters of diphenylboric acid: antimicrobial activity

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10085368.2
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Page 262

- AU Molodykh, Zh. V.; Anisimova, N. N.; Nikonov, G. N.; Erastov, O. A.
- CS Inst. Org. Fiz. Khim. im. Arbuzova, Kazan, USSR
- SO Khimiko-Farmatsevticheskii Zhurnal (1988), 22(4), 438-41 CODEN: KHFZAN; ISSN: 0023-1134
- DT Journal
- LA Russian
- IT 2622-89-1DP, glycolic esters

RL: BAC (Biological activity or effector, except adverse); BSU (Biological study, unclassified); SPN (Synthetic preparation); BIOL (Biological study); PREP (Preparation)

(prepn. and antimicrobial activity of, structure in relation to)

- RN 2622-89-1 CAPLUS
- CN Borinic acid, diphenyl- (6CI, 7CI, 8CI, 9CI) (CA INDEX NAME)

- AB Glycolic diphenylboric esters with the general formula of HOROBPh2 and complexes with HORB (.rarw.A)PH2 amines were synthesized and studied for their toxicity against fungi, bacteria, white mice, and the lipolytic enzyme lipase. The introduction of an amine fragment into the mol. of glycolic di-Ph ester diminished toxicity in warm-blooded animals and suppressed the activity of lipase to a greater extent. The aliph. radical length at the nitrogen atom exerted significant effects on the growth of Trichophyton and bacteria. Substantially augmented antimicrobial properties were assocd. with the greater lipophility of the compds., and it was suggested that the compds. might act in the open tautomeric form.
- L4 ANSWER 177 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN
- AN 1988:427375 CAPLUS
- DN 109:27375
- TI Preconcentration and analysis of tetraphenylboron and diphenylborinic acid in natural waters using C18 reverse-phase liquid chromatography
- AU Mills, Gary L.; Schwind, Donna; Adriano, Domy C.
- CS Inst. Ecol., Univ. Georgia, Aiken, SC, 29801, USA
- SO Chemosphere (1988), 17(5), 937-42 CODEN: CMSHAF; ISSN: 0045-6535
- DT Journal
- LA English
- IT 2622-89-1, Diphenylborinic acid

RL: ANT (Analyte); ANST (Analytical study)

(detn. of, in natural water, by reversed-phase HPLC)

- RN 2622-89-1 CAPLUS
- CN Borinic acid, diphenyl- (6CI, 7CI, 8CI, 9CI) (CA INDEX NAME)

AB Tetraphenylboron (TPB) and diphenylborinic acid (DPBA) were concd. from aq. solns. using C18 reverse-phase liq. chromatog. Percent recoveries from solns. contg. 0.05-1.199 .mu.g/mL were 94.6% and 82.8% for TPB and DPBA, resp. The effect of soln. vol. was also investigated and the

results indicated that .ltoreq.50 mL of sample can be processed through a column without significant loss in recovery efficiency. Naturally occurring dissolved org. matter present in a black water coastal plain stream did not interfere in the retention or elution efficiencies for these organoborates.

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L4 ANSWER 178 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN
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AN 1988:217490 CAPLUS

DN 108:217490

TI Response of loblolly pine (Pinus taeda L.) seedlings to soil-applied organo-borates

AU Kaplan, D. I.; Burkman, W. G.; Adriano, D. C.

CS Biogeochem. Div., Savannah River Ecol. Lab., Aiken, SC, 29801, USA

SO Water, Air, and Soil Pollution (1988), 37(1-2), 73-83 CODEN: WAPLAC; ISSN: 0049-6979

DT Journal

LA English

IT 2622-89-1

RL: BIOL (Biological study)

(tetraphenylborate degrdn. product, loblolly pine seedlings response to soil-applied)

RN 2622-89-1 CAPLUS

CN Borinic acid, diphenyl- (6CI, 7CI, 8CI, 9CI) (CA INDEX NAME)

AB Two greenhouse pot expts. using loblolly pine (P. taeda) seedlings were conducted to evaluate the effects of Na tetraphenylboron (NaTPB) and one of its degrdn. byproducts, diphenylboric acid (DPBA), on pine B nutrition and growth. The needle and root tissue concns. of B were higher for NaTPB than DPBA treatments. Consequently, NaTPB but not DPBA had detrimental effects on plant growth. Seedlings that had significant yield detriments displayed typical B toxicity symptoms due to high-B stress. The distribution of B among the needles, stems and roots, expressed as percent of total B in the seedlings, remained relatively const. irresp. of the soil B level or B source. The peak of hot-water extractable soil B from the NaTPB treatments lagged about 20 days behind the DPBA treatments, suggesting a faster hydrolysis for the latter compd.

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L4 ANSWER. 179 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN
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AN 1988:112528 CAPLUS

DN 108:112528

TI Structural studies of organoboron compounds. XXV. Synthesis and structure of (maltolato)diphenylboron

AU Orvig, Chris; Rettig, Steven J.; Trotter, James

CS Dep. Chem., Univ. British Columbia, Vancouver, BC, V6T 1Y6, Can.

SO Canadian Journal of Chemistry (1987), 65(3), 590-4 CODEN: CJCHAG; ISSN: 0008-4042

DT Journal

LA English

OS CASREACT 108:112528

IT 2622-89-1P, Diphenylborinic acid RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)

Patel 9/24/2003>

(prepn. and reaction of, with maltol)

RN 2622-89-1 CAPLUS

CN Borinic acid, diphenyl- (6CI, 7Cİ, 8CI, 9CI) (CA INDEX NAME)

Ph | Ph— B— OH

AB The reaction of maltol (3-hydroxy-2-methyl-4-pyrone) with diphenylborinic acid gave 90% of the title compd. Its crystal structure was detd. The mol. contains a 5-membered C2O2B ring having a flattened B-envelope conformation, the B atom being displaced 0.081(2) .ANG. from the C2O2 plane. Structural and spectroscopic data are consistent with weak binding of the maltolate O atoms to B.

L4 ANSWER 180 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

AN 1988:18588 CAPLUS

DN 108:18588

TI Effect of hydroxyorganoboranes on synthesis, transport and N-linked glycosylation of plasma proteins

AU Goldberger, Gabriel; Paz, Mercedes A.; Torrelio, B. Marina; Okamoto, Yoshiaki; Gallop, Paul M.

CS Harvard Sch. Med., Child. Hosp. Corp., Boston, MA, 02115, USA

SO Biochemical and Biophysical Research Communications (1987), 148(1), 493-9 CODEN: BBRCA9; ISSN: 0006-291X

DT Journal

LA English

IT 2622-89-1

RL: ANST (Analytical study)

(inhibition by, of protein formation and transport and glycosylation in human cells)

RN 2622-89-1 CAPLUS

CN Borinic acid, diphenyl- (6CI, 7CI, 8CI, 9CI) (CA INDEX NAME)

AB By using a recently developed method (Boradeption) for transfering water-insol. hydroxyorganoborane compds. into cells, inhibition of protein synthesis by 3 of these compds. and inhibition of secretion of plasma proteins by 4 of them were obsd. in human hepatoma HepG2 cells. These effects were specific in that the cell viability was not affected and an increase in protein catabolism was not obsd. Three compds. caused compd.-specific alterations in the electrophoretic mobility of secreted glycoproteins due to underlying changes in the N-linked carbohydrate moieties. Results presented suggest a potential new source of cellular probes.

L4 ANSWER 181 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

AN 1987:469592 CAPLUS

DN 107:69592

TI Synthesis and spectroscopic and structural characterization of derivatives of the quasi-alkoxide ligand [OBMes2]- (Mes = 2,4,6-Me3C6H2)

AU Weese, Kenneth J.; Bartlett, Ruth A.; Murray, Brendan D.; Olmstead, Marilyn M.; Power, Philip R.

CS Dep. Chem., Univ. California, Davis, CA, 95616, USA

SO Inorganic Chemistry (1987), 26(15), 2409-13

CODEN: INOCAJ; ISSN: 0020-1669

DT Journal

LA English

IT 20631-84-9

RL: PRP (Properties) (crystal structure of)

RN 20631-84-9 CAPLUS

CN Borinic acid, bis(2,4,6-trimethylphenyl) - (9CI) (CA INDEX NAME)

Treatment of Mes2BOH; (Mes = 2,4,6-Me3C6H2) with BuLi in hexane/ether affords a suspension of LiOBMes2, which can be crystd. from THF soln. as the dimer [{Li(THF)OBMes2}2] (II). Treatment of a slurry of anhyd. CoCl2 in THF with 2 equiv. of II gives [Co{OBMes2}2Li(THF)2Cl2Li(THF)2] (III) in good yield. The x-ray crystal structures of I, II, and III are also reported. The structure of I is the 1st for a diorganoboronous acid, and it exists in the solid state as H-bonded tetramers. II is the 1st structurally characterized example of a metal salt of a boronous acid, and it possesses a dimeric structure previously seen only with very bulky -OC(tert-Bu)3 and -OC6H2-2,6-tert-Bu2-4-Me salts. III has Co pseudotetrahedrally bound to 2 OBMes2 and Cl- ligands, which also form bridges to 2 Li+ ions. Each Li+ is also pseudotetrahedrally coordinated, with 2 THF donors as the remaining ligands in each case.

L4 ANSWER 182 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

AN 1986:207334 CAPLUS

DN 104:207334

TI Studies on antitumor boron containing compounds. IV. Synthesis and antitumor activities of bis(p-methoxyphenyl)borinic .alpha.-amino acid anhydrides

AU Lin, Kai; Zhang, Guomin; Fu, Naiwu

CS Dep. Chem., Wuhan Univ., Wuhan, Peop. Rep. China

SO Youji Huaxue (1985), (3), 228-32 CODEN: YCHHDX; ISSN: 0253-2786

DT Journal

LA Chinese

Patel

OS CASREACT 104:207334

IT 73774-45-5P

RL: SPN (Synthetic preparation); PREP (Preparation) (prepn. and reactions with hydroxyethylamine and amino acids)

RN 73774-45-5 CAPLUS

CN Borinic acid, bis(4-methoxyphenyl) - (9CI) (CA INDEX NAME)

9/24/2003>

The title compds. (p-MeOC6H4)2BO2CCHRNH2 I [R = H, Me, CHMe2, CH2CHMe2, CH2Ph, (CH2)2SMe, 3-indolylmethyl, 3-bis(p-methoxyphenyl)boryl-4-imidazolylmethyl] were prepd. by the reactions of (p-MeOC6H4)2BOH, obtained from Grignard reaction of p-MeOC6H4Br with B(OBu)3, with DL-, D-, or L-H2NCHRCO2H in PhMe. Preliminary tests showed D-I (R = CH2CHMe2) had 36.4% inhibitory activity on Ehrlich ascites carcinoma cells in mice at 0.4 g/kg and D-I (R = CH2CH2SMe) had 49.5% inhibitory activity at 0.6 g/kg.

L4 ANSWER 183 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

AN 1985:472876 CAPLUS

DN 103:72876

TI Fluorochlorohydrocarbon compositions

IN Enjo, Naonori; Harada, Yuhkow

PA Daikin Kogyo Co., Ltd., Japan

SO Eur. Pat. Appl., 28 pp.

CODEN: EPXXDW

DT Patent

LA English

FAN CNT 1

1	AN. CNI I				
	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
]	PI EP 136683	A2	19850410	EP 1984-111678	19840929
	EP 136683	A3	19860326		
	EP 136683	B1 ·	19911204		
	R: DE, FR,	GB			
				JP 1983-183870	19830930
,	JP 60072979	A2	19850425	JP 1983-183870	19830930
	JP 03069956	B4	19911105		
	US 4623475	A	19861118	US 1984-655527	19840928
				JP 1983-183870	19830930

IT 2622-89-1

RL: MOA (Modifier or additive use); USES (Uses) (heat stabilizers, for chlorofluorocarbons)

RN 2622-89-1 CAPLUS

CN Borinic acid, diphenyl- (6CI, 7CI, 8CI, 9CI) (CA INDEX NAME)

AB The title compns. contain a B compd. or a B compd. and an ester of phosphorous acid in order to inhibit decompn. of the halocarbon compd. at high temps. The stabilized compns. are useful as heating fluids and as foaming agents for plastics. Thus, 2 g Cl3CF [75-69-4] contg. 0.25% triphenyl borate (I) [1095-03-0] was placed in a glass tube along with 0.02 g lubricating oil (turbine oil) and a piece of steel. After the tube was sealed and heated 100 h at 150.degree., the liq. contained 15 ppm Cl, compared with 1500 ppm without I. The appearance of the liq. and the steel did not change during heating, while heating in the absence of I

Patel 9/24/2003>

caused discoloration of the liq. and steel.

L4 ANSWER 184 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

AN 1985:422778 CAPLUS

DN 103:22778

TI Syntheses and reactions of pyrazaboles

AU Layton, W. J.; Niedenzu, Kurt; Niedenzu, P. M.; Trofimenko, S.

CS Dep. Chem., Univ. Kentucky, Lexington, KY, 40506, USA

SO Inorganic Chemistry (1985), 24(10), 1454-7 CODEN: INOCAJ; ISSN: 0020-1669

DT Journal

LA English

OS CASREACT 103:22778

IT 12113-07-4

RL: RCT (Reactant); RACT (Reactant or reagent)
 (reaction of, with ammonia)

RN 12113-07-4 CAPLUS

CN Borate(1-), hydroxytriphenyl-, sodium, (T-4)- (9CI) (CA INDEX NAME)

Na +

GI

$$N-N$$
 $N-N$
 $N-N$
 $N-N$
 $N-N$
 $N-N$
 $N-N$

AB The unsym. pyrazabole Ph2B(.mu.-pz)2BH2 (I, pz = N2C3H3 = pyrazolyl) was prepd. by the reaction of K[Ph2B(pz)2] with Me3NBH2I; subsequent halogenation with Br2 yielded Ph2B(.mu.-pz)2BBr2. Similarly, Et2B(.mu.-pz')2BH2 (Hpz' = 3,5-dimethylpyrazole) was converted to Et2B(.mu.-pz')2BBr2. Reaction of H2B(.mu.-pz')2BH2 with (even an excess of) BBr3 gave a mixt. of cis and trans isomers of HBrB(.mu.-pz')2BHBr, whereas reaction with Br2 afforded Br2B(.mu.-pz')2BBr2. Reaction of the latter compd. with K[pz] yielded (pz)2B(.mu.-pz')2B(pz)2, the first characterized pyrazolylpyrazabole contg. different pyrazolyl moieties

bonded to the same B atom. A second, polymeric modification of Hpz'B(.mu.-pz')2BHpz' was identified; it appears to be the one reacting with addnl. Hpz' to form (pz')2B(.mu.-pz')2B(pz')2. The latter forms a monohydrate in a reversible reaction, but no similar interaction occurs with NH3.

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ANSWER 185 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN
L4
AN
    1985:204099 CAPLUS
DN
    102:204099
ΤI
    Recovery of arylboranes
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Ostermaier, John Joseph IN

du Pont de Nemours, E. I., and Co. , USA PΑ

SO Eur. Pat. Appl., 10 pp. CODEN: EPXXDW

DT Patent

English LA

FAN.CNT 1

11111.											′		
	PATENT NO.			KIND	DATE	DATE		APPLICATION NO.			DATE		
			-										
ΡI	ΕP	1313	07		A2	1985	0116		ΕP	1984-10810)2	19840	711
	EP	13130	07		A 3	1985	1227						
	ΕP	1313	07		B1	1989	0503						•
		R:	BE,	DE,	FR, GE	3, IT,	LU,	NL					
									US	1983-51268	36	19830	711
	US	4521	628		A	1985	0604		US	1983-51268	36	19830	711
	CA	1201	728		A1	1986	0311		CA	1984-45848	39	19840	710
									US	1983-51268	36	19830	711
	JP	60038	8388		A2	1985	0227		JΡ	.1984-14248	34	19840	711
	JP	0500	3473		B4	1993	0114						
									US	1983-51268	36	19830	711

ΙT 2622-89-1

RL: RCT (Reactant); RACT (Reactant or reagent)

(neutralization of tetraphenylborane adduct in presence of)

RN 2622-89-1 CAPLUS

CN Borinic acid, diphenyl- (6CI, 7CI, 8CI, 9CI) (CA INDEX NAME)

12113-07-4 IT

RL: RCT (Reactant); RACT (Reactant or reagent) (neutralization of, with hydrochloric acid, triphenylborane from)

RN 12113-07-4 CAPLUS

Borate(1-), hydroxytriphenyl-, sodium, (T-4)- (9CI) (CA INDEX NAME) CN

Na +

AB Arylboranes were recovered from their basic adducts in aq. soln. by neutralization in at least 2 stages. Thus, a mixt. of 0.10 phenylborinic acid, 0.25 diphenylborinic acid, 8.5 Ph3B, 0.5 tetraphenylborate, 4.2 NaOH, 80.0 H2O, and 6.2 wt. % NaCl was neutralized in 2 stages using aq. 7% HCl at 40.degree..

L4 ANSWER 186 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

AN 1985:163282 CAPLUS

DN 102:163282

TI Mass spectral and HPLC analysis of biological compounds with diphenylborinic acid

AU Flueckiger, Rudolf; Henson, Edward; Hess, Guido M.; Gallop, Paul M.

CS Child. Hosp., Harvard Sch. Med., Boston, MA, 02115, USA

SO Biomedical Mass Spectrometry (1984), 11(12), 611-15 CODEN: BMSYAL; ISSN: 0306-042X

DT Journal

DT Journal

LA English

IT 2622-89-1

RL: ANST (Analytical study)

(derivatization by, of biomols. for HPLC and mass spectrometry)

RN 2622-89-1 CAPLUS

CN Borinic acid, diphenyl- (6CI, 7CI, 8CI, 9CI) (CA INDEX NAME)

AB Diphenylborinic acid is shown to react with a variety of polyfunctional mols. of biol. interest (e.g., amino acids) to give products which are easily identifiable by mass spectrometry. The diphenylborinate adducts of amino acids possess extraordinary stability and are separable by reversed-phase HPLC. The stability of products is discussed with regard to the nature and stereochem. arrangement of the functional groups involved in complex formation.

L4 ANSWER 187 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

AN 1985:62296 CAPLUS

DN 102:62296

TI Synthesis of diaryl- and arylboric acids

AU Sazonova, E. V.; Galiullina, R. F.

CS USSR

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Khimiya Elementoorganicheskikh Soedinenii (1983) 24-5
SO
     CODEN: KELSDE; ISSN: 0201-6699
DT
    Journal
LΑ
     Russian
     CASREACT 102:62296
OS
IT
     2622-89-1P
     RL: SPN (Synthetic preparation); PREP (Preparation)
        (prepn. of)
     2622-89-1 CAPLUS
RN
     Borinic acid, diphenyl- (6CI, 7CI, 8CI, 9CI) (CA INDEX NAME)
CN
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Ph | Ph— B— OH

AB Treating NaBPh4 with HCl in dioxane gave 90% Ph2BOH. KB(C6H4Me-p)4 and HCl gave 35% p-MeC6H4B(OH)2.

L4 ANSWER 188 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN
AN 1985:42122 CAPLUS
DN 102:42122
TI Serum cholinesterase inhibition by boronic acids
AU Garner, Charles W.; Little, Gwynne H.; Pelley, John W.
CS Health Sci. Cent., Texas Tech Univ., Lubbock, TX, 79430, USA

SO Biochimica et Biophysica Acta (1984), 790(1), 91-3 CODEN: BBACAQ; ISSN: 0006-3002

LA English
IT 2622-89-1
RL: BIOL (Biological study)
(cholinesterase inhibition by, kinetics of)

RN 2622-89-1 CAPLUS CN Borinic acid, diphenyl- (6CI, 7CI, 8CI, 9CI) (CA INDEX NAME)

Ph | Ph— B— OH

DT

Journal

AB Horse serum cholinesterase (EC 3.1.1.8) was reversibly inhibited by a variety of alkyl- and areneboronic acids with Ki values ranging from 6.2 mM (methaneboronic acid) to 3.1 .mu.M (diphenylboric acid). Binding to the enzyme was apparently at the active center, because inhibition obeyed competitive kinetics and because boronic acids protected the enzyme from inactivation by phenylmethanesulfonyl fluoride. Boronic acids should prove useful in probing the active center of serum cholinesterase.

- L4 ANSWER 189 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN
- AN 1984:447599 CAPLUS
- DN 101:47599
- TI Coordination chemistry of copper macrocyclic complexes: synthesis and characterization of copper complexes of TIM
- AU Maroney, Michael J.; Rose, Norman J.
- CS Dep. Chem., Univ. Washington, Seattle, WA, 98195, USA
- SO Inorganic Chemistry (1984), 23(15), 2252-61

10085368.2

Page 271

CODEN: INOCAJ; ISSN: 0020-1669

DTJournal

LΑ English

ΙT 90342-86-2P

RL: SPN (Synthetic preparation); PREP (Preparation)

(prepn. of)

RN

90342-86-2 CAPLUS
Copper(1+), [hydroxytriphenylborato(1-)](2,3,9,10-tetramethyl-1,4,8,11-CN tetraazacyclotetradeca-1,3,8,10-tetraene-.kappa.N1,.kappa.N4,.kappa.N8,.ka ppa.N11)-, (SP-5-12)-, tetraphenylborate(1-) (9CI) (CA INDEX NAME)

CM

CRN 90342-85-1

CMF C32 H40 B Cu N4 O

· CCI CCS

CM

CRN 4358-26-3

CMF C24 H20 B

CCI CCS

GI

AB Four-coordinate planar [Cu(TIM)] (BPh4)2(TIM = I) and 5-coordinate pyramidal [Cu(TIM)L](PF6)2 (L = 1-methylimidazole, imidazole, py, NH3) and [Cu(TIM)X]PF6 (X = Cl, Br, I, NCS) were prepd. The complexes were characterized in the solid state and in the soln. by elemental anal., IR, electronic, and EPR spectroscopy, and magnetic, cond., and mol. wt. measurements. The position of PF6- in the solid-state structures of [Cu(TIM)X]PF6 (X = Cl, Br) is such that the displacement of Cu from the basal ligand plane in these pyramidal structures is smaller than might be expected. In Me2CO soln., [Cu(TIM)](BPh4)2 undergoes a redn. to the intensely blue Cu(TIM)+ (Emax = 1.34 .mu.m-1). In the presence of excess NCS-, Cu(TIM)NCS+ in Me2CO soln. exists in equil. with the 6-coordinate Cu(TIM)(NCS)2.

L4ANSWER 190 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

AN 1984:209930 CAPLUS

DN 100:209930

ΤI The reactions of diborane with aryl organotin compounds

ΑU Pickles, G. M.; Spencer, T.; Thorpe, F. G.; Chopa, A. B.; Podesta, J. C.

CS Chem. Dep., Univ. Lancaster, LA1 4YA, UK

SO Journal of Organometallic Chemistry (1984), 260(1), 7-15 CODEN: JORCAI; ISSN: 0022-328X

DTJournal

LΑ English

OS CASREACT 100:209930

2622-89-1P 66117-64-4P 89566-59-6P IT

RL: SPN (Synthetic preparation); PREP (Preparation)

(prepn. of, by hydrolysis of aryltin-borane reaction product)

2622-89-1 CAPLUS RN

CN Borinic acid, diphenyl- (6CI, 7CI, 8CI, 9CI) (CA INDEX NAME)

RN 66117-64-4 CAPLUS

CN Borinic acid, bis(4-methylphenyl) - (9CI) (CA INDEX NAME)

89566-59-6 CAPLUS RN

CN Borinic acid, bis(4-chlorophenyl) - (9CI) (CA INDEX NAME)

AB Treating R4Sn (R = Ph, 2- and 4-tolyl, 4-ClC6H4) and Ph3SnX (X = Cl, H, OH, OAc, O2CCF3) with BH3 gave transmetalation, in which .gtoreq.1 aryl group was transferred to B. The organoboron intermediates give phenols upon oxidn. and boronic and borinic acids upon hydrolysis. Pyridine (L) complexes of organoboranes, Ph2BHL and Ph3BL were also isolated.

L4 ANSWER 191 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

1984:192221 CAPLUS ΑN

DN 100:192221

ΤI Synthesis of bis(p-methoxyphenyl)borinic .alpha.-amino acid anhydrides

ΑU Lin, Kai; Zhang, Guomin

CS

Wuhan Univ., Wuhan, Peop. Rep. China Ziran Zazhi (1983), 6(9), 715-17 SO CODEN: TJTCD4; ISSN: 0253-9608

DT Journal

LΑ Chinese

IT 73774-45-5P

RL: SPN (Synthetic preparation); PREP (Preparation)

(prepn. and condensation with amino acids and aminoethanol)

RN 73774-45-5 CAPLUS

CNBorinic acid, bis(4-methoxyphenyl) - (9CI) (CA INDEX NAME)

GΙ

AB Title borinic amino acid anhydrides I (R = amino acid residue) were prepd. by reaction of (p-MeOC6H4)2BOH with H2NCHRCO2H (NHCHRCO = Val, Phe, Tyr, Leu, Met, Ala, etc.).

L4 ANSWER 192 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

AN 1984:6728 CAPLUS

DN 100:6728

TI Organozinc derivatives of dialkyl(aryl)borinic acids

AU Galiumena, R. F.; Sazonova, E. V.; Dodonov, V. A.

Ι

CS USSR

SO Khimiya Elementoorgan. Soedin., Gor'kii (1982) 33-6 From: Ref. Zh., Khim. 1983, Abstr. No. 14Zh278

DT Journal

LA Russian

IT 62981-91-3

RL: RCT (Reactant); RACT (Reactant or reagent)
 (reaction of, with dialkylzinc)

RN 62981-91-3 CAPLUS

CN Borinic acid, di-1-naphthalenyl- (9CI) (CA INDEX NAME)

AB Title only translated.

L4 ANSWER 193 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

AN 1983:595137 CAPLUS

DN 99:195137

TI Organozinc derivatives of dialkyl(aryl)boric acids

AU Galiullina, R. F.; Sazonova, E. V.; Dodonov, V. A.

CS Gor'k. Gos. Univ., Gorkiy, USSR

SO Khimiya Elementoorganicheskikh Soedinenii (1982) 33-6 CODEN: KELSDE; ISSN: 0201-6699

DT. Journal

LA Russian

OS CASREACT 99:195137

IT 62981-91-3

RL: RCT (Reactant); RACT (Reactant or reagent)
 (reaction of, with zinc compds.)

RN 62981-91-3 CAPLUS

CN Borinic acid, di-1-naphthalenyl- (9CI) (CA INDEX NAME)

AB RZnOBR12 (I, R = Et, isopentyl, R1 = Bu; R = Et, R1 = .alpha.-naphthyl) were prepd. by treating R2Zn with R12BuOH. Some reactions of I were discussed. I (R = Et, R1 = Bu) is an effective catalyst for the copolymn. of ethylene oxide with maleic anhydride.

L4 ANSWER 194 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

AN 1983:585388 CAPLUS

DN 99:185388

TI Structural studies of organoboron compounds. XV. Crystal and molecular structures of (3-aminopropanolato)diphenylboron, (2-N,N-dimethylaminoethanolato)diphenylboron, and (2-N,N-dimethylaminoethanolato)diphenylboron-diphenylborinic acid (1:1)

AU Rettig, Steven J.; Trotter, James

CS Dep. Chem., Univ. British Columbia, Vancouver, BC, V6T 1Y6, Can.

SO Canadian Journal of Chemistry (1983), 61(10), 2334-40 CODEN: CJCHAG; ISSN: 0008-4042

DT Journal

LA English

IT **87654-46-4 87699-91-0**RL: PRP (Properties)
(structure of)

RN 87654-46-4 CAPLUS

CN Boron, [2-(dimethylamino)ethanolato-N,O]diphenyl-, (T-4)-, compd. with diphenylborinic acid (1:1) (9CI) (CA INDEX NAME)

CM 1

CRN 87654-45-3 CMF C16 H20 B N O CCI CCS

CM 2

CRN 2622-89-1 CMF C12 H11 B O

Ph | Ph— B— OH

RN 87699-91-0 CAPLUS

CN Borinic acid, diphenyl-, compd. with 2-(dimethylamino)ethyl diphenylborinate (1:1) (9CI) (CA INDEX NAME)

CM 1

CRN 2622-89-1, CMF C12 H11 B O

Ph | | | Ph— B— OH

CM 2

CRN 1028-93-9 CMF C16 H20 B N O

 $\begin{array}{c} \text{Ph} \\ | \\ \text{Ph-B-O-CH}_2\text{--CH}_2\text{--NMe}_2 \end{array}$

AB (3-Aminopropanolato)diphenylboron is monoclinic, space group P21/n, with a 9.6717(13), b 9.8867(6), c 14.452(2) .ANG., and .beta. 99.500(7).degree.; Z = 4. (2-N,N-Dimethylaminoethanolato)diphenylboron is monoclinic, space group Cc, with a 7.0721(4), b 16.8829(4), c 12.0975(8) .ANG., and .beta. 97.875(3).degree.; Z = 4. (2-N,N-Dimethylaminoethanolato)diphenylboron - diphenylborinic acid (1:1) is monoclinic, space group P21/n, with a 11.3231(10), b 19.3190(12), c 12.2451(11), .ANG., and .beta. 109.321(4); Z = 4. All 3 structures were solved by direct methods and refined by full-matrix least-squares to final R1 of 0.038, 0.031, and 0.040, resp. Each structure contains a tetrahedrally coordinated B. The libration-cor. B-O, B-N, and mean B-C distances are: 1.481(2), 1.643(3), and 1.623(3) .ANG. for (aminopropanolato)diphenylboron; 1.476(2), 1.691 (2), and 1.625(7) .ANG. for (dimethylaminoethanolato)diphenylboron. The Ph2BOH mol. contains a trigonal-planar B atom with B-O 1.354(3) .ANG. and mean B-C 1.572(3) .ANG.

L4 ANSWER 195 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

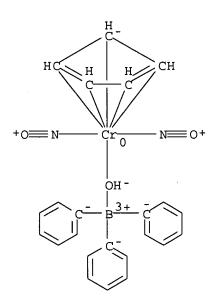
AN 1983:505428 CAPLUS

DN 99:105428

9/24/2003>

- Organometallic nitrosyl chemistry. 19. Protonation vs. oxidative cleavage of the isoelectronic complexes [(.eta.5-C5H4R)M(LO)2]2 (M = Cr, Mn, or Fe; L = C or N; R = H or Me) by HBF4
- AU Legzdins, Peter; Martin, David T.; Nurse, Charles R.; Wassink, Berend
- CS Dep. Chem., Univ. British Columbia, Vancouver, BC, V6T 1Y6, Can.
- SO Organometallics (1983), 2(9), 1238-44 CODEN: ORGND7; ISSN: 0276-7333
- DT Journal
- LA English
- IT 86365-56-2P

- RN 86365-56-2 CAPLUS
- CN Chromium, (.eta.5-2,4-cyclopentadien-1-yl)[hydroxytriphenylborato(1-)]dinitrosyl- (9CI) (CA INDEX NAME)



Treatment of [(.eta.5-C5H5)Fe(CO)2]2 with an equimolar amt. of AB HBF4.cntdot.OMe2 in CH2Cl2 results in the clean formation of [(.eta.5-C5H5)2Fe2(CO)4H]BF4 which may be isolated in good yield. contrast, 2 equiv of the acid are required to consume completely [(.eta.5-C5H5)Cr(NO)2]2, the principal organometallic product being (.eta.5-C5H5)Cr(NO)2BF4. This latter complex is not isolable, but it can be identified spectroscopically and by its deriv. chem. Some of the workup procedures employed also afford new organometallic nitrosyl complexes of Cr such as $[{(.eta.5-C5H5)Cr(NO)2}2OH]BF4$ and (,eta.5-C5H5)Cr(NO)2(OHBPh3). Two equiv. of HBF4.cntdot.OMe2 also consume [(.eta.5-C5H4R)Mn(CO)(NO)]2 (R = H, Me), but a complex mixt. of products results. Two well-known (i.e. [(.eta.5-C5H4R)Mn(CO)2(NO)]+ and (.eta.5-C5H4R)2Mn2(NO)3(NO2)) and 2 novel (i.e., [(.eta.5-C5H4R)3Mn3(NO)3NH]+ and [(.eta.5-C5H4R)2Mn2(NO)2(CO)(NH2)]+) types of Mn nitrosyl complexes are produced in each case, the novel cations being ultimately isolable in low yields as the BF4- and BPh4- salts, resp. Cyclic voltammograms of [(.eta.5-C5H5)Fe(CO)2]2 and [(.eta.5-C5H5)Cr(NO)2]2 recorded under identical exptl. conditions reveal that the Cr dimer undergoes oxidn. at a slightly more pos. potential. The propensities of the [(.eta.5-C5H4R)M(LO)2]2 (M = Cr, Mn, Fe; L = C, N; R =

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H, Me) dimers to undergo protonation or oxidative cleavage when treated with H+ are thus rationalized in terms of the stabilities of the initially formed [(.eta.5-C5H4R)M(LO)2]2H+ adducts. Interestingly, treatment of [(.eta.5-C5H5)Co(NO)]2 with HBF4.cntdot.OMe2 in CH2Cl2 results in simple oxidn., the known [(.eta.5-C5H5)Co(NO)]2BF4 complex being obtainable in good yield.

- L4 ANSWER 196 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN
- AN 1983:488250 CAPLUS
- DN 99:88250
- TI Dioxazaboronines
- AU Moehrle, Hans; Zuege, Erika; Bluhme-Hensen, Karin
- CS Inst. Pharm. Chem., Univ. Duesseldorf, Duesseldorf, D-4000/1, Fed. Rep. Ger
- SO Archiv der Pharmazie (Weinheim, Germany) (1983), 316(4), 289-97 CODEN: ARPMAS; ISSN: 0365-6233
- DT Journal
- LA German
- IT 2622-89-1

RL: RCT (Reactant); RACT (Reactant or reagent)
 (reaction of, with naphthoxazinylnaphthols)

RN 2622-89-1 CAPLUS

CN Borinic acid, diphenyl- (6CI, 7CI, 8CI, 9CI) (CA INDEX NAME)

GΙ

- AB Treating H2NCH2CH2OBPh2 with phenols and H2CO led, depending on the phenol, either to B chelates of Mannich bases, e.g., I, or with extrusion of benzene to II (R = Me, R1 = CMe3, CMeEt2; R = CMe3, R1 = Me).
- L4 ANSWER 197 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN
- AN 1983:143499 CAPLUS
- DN 98:143499
- TI Structural studies of organoboron compounds. XIII. Preparation and crystal and molecular structure of bis[(salicylaldoximato(2-))phenylboron]
- AU Rettig, Steven J.; Trotter, James
- CS Dep. Chem., Univ. British Columbia, Vancouver, BC, V6T 1Y6, Can.
- SO Canadian Journal of Chemistry (1983), 61(1), 206-10 CODEN: CJCHAG; ISSN: 0008-4042

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DT Journal
LA English
IT 2622-89-1
RL: RCT (Reactant); RACT (Reactant or reagent)
(reaction of, with salicylaldoxime)
RN 2622-89-1 CAPLUS
CN Borinic acid, diphenyl- (6CI, 7CI, 8CI, 9CI) (CA INDEX NAME)

AB The reaction of Ph2BOH with salicylaldoximme yielded [(C7H5NO2)BPh]2 (I) via a B-C bond cleavage reaction. The crystal structure of I indicated that the system has five fused 6-membered rings including the first crystallog. characterized B2N2O2 ring. I is stabilized both by intramol. N .fwdarw. B coordination and by resonance delocalization and contains B atoms in a distorted tetrahedral environment.

L4 ANSWER 198 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

AN 1983:47062 CAPLUS

DN 98:47062

TI Liquid-liquid extraction systems for the isolation of catecholamines from serum and urine for HPLC analysis

AU Smedes, Foppe; Kraak, Johan C.; Poppe, Hans

CS Lab. Anal. Chem., Univ. Amsterdam, Amsterdam, 1018 WV, Neth.

SO Proc. - Int. Congr. Clin. Chem., 11th (1982), Meeting Date 1981, 977-81. Editor(s): Kaiser, Erich; Gabl, Franz; Mueller, Mathias M. Publisher: de Gruyter, Berlin, Fed. Rep. Ger.

CODEN: 48YDAZ

DT Conference

LA English

IT 83075-94-9
RL: BIOL (Biological study)

(in catecholamine extn. from human biol. ligs., for chromatog.)

RN 83075-94-9 CAPLUS

CN Borate(1-), dihydroxydiphenyl-, (T-4)- (9CI) (CA INDEX NAME)

Liq.-liq. extn. of the catecholamines is based on the formation of a complex between H3BO3 or organoboric acids and diol groups of adrenaline [51-43-4], noradrenaline [51-41-2], and dopamine [51-61-6]. Hydrophobic substituents of diphenylborate [83075-94-9] used in the extn. favors the extn. of the borate-diol complex into org. solvents. To det. optimal extg. conditions, the effects of distribution coeff., pH, type of pairing ion, diphenylborate concn., etc., on the extn. procedures were examd. The extn. procedure involves shaking (10 min) of 1 mL plasma, 2 mL 1M NH4OH/NH4Cl buffer (pH 8.9) contg. 0.1% com. diphenylborate-ethanolamine [15614-89-8] plus 2 mL H2O (contg. the internal std.) with 5

mL hexane plus 1% n-octanol contg. 0.3% (C8H17)4N+Br-. The back extn. involved addn. of 1-2 mL n-octanol, 0.4-1 mL AcOH; the mixt. is then shaken and the aq. phase analyzed. The recovery of catecholamines from spiked urine and plasma samples was 94-100%.

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L4 ANSWER 199 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN
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AN 1982:538760 CAPLUS

DN 97:138760

TI Simple and fast solvent extraction system for selective and quantitative isolation of adrenaline, noradrenaline and dopamine from plasma and urine

AU Smedes, F.; Kraak, J. C.; Poppe, H.

CS Lab. Anal. Chem., Univ. Amsterdam, Amsterdam, 1018 WV, Neth.

SO Journal of Chromatography (1982), 231(1), 25-39 CODEN: JOCRAM; ISSN: 0021-9673

DT Journal

LA English

IT 83075-94-9

RL: BIOL (Biological study)

(in catecholamine extn. from blood plasma and urine)

RN 83075-94-9 CAPLUS

CN Borate(1-), dihydroxydiphenyl-, (T-4)- (9CI) (CA INDEX NAME)

A solvent extn. system for the selective and quant. isolation of AΒ adrenaline [51-43-4], noradrenaline [51-41-2] and dopamine [51-61-6] from plasma and urine is described. The extn. system makes use of the complex formation, in alk. medium, between diphenylborate [83075-94-9] and the diol group in the catecholamines in combination with ion-pair formation. The influence of various parameters on the distribution coeff. was investigated by anal. of the liq. phases by high-performance liq. chromatog. with electrochem. detection for selecting the optimal extn. conditions. With hexane + 1% octanol contq. 0.25% of tetraoctylammonium bromide as the extn. solvent, the catecholamines can be quant. isolated from plasma and urine at pH 8.6 in the presence of 0.1% of diphenylborate. For urine the recovery was 101.5 for adrenaline, 100.6% for noradrenaline and 99.9% for dopamine. For plasma the recoveries were, resp., 101.8%, 100.5% and 92.9%. The recovery of dihydroxybenzylamine, included in the study as internal std., was 96.3% for urine and 89.9% for plasma. The applicability of the developed extn. system as clean-up and concn. step for the anal. of catecholamines in plasma and urine by high-performance liq. chromatoq. with electrochem. detection is demonstrated.

- L4 ANSWER 200 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN
- AN 1982:527689 CAPLUS
- DN 97:127689
- TI Dimesitylboryl compounds. Part 8. Bis(dimesitylboryl)methane
- AU Garad, Manchak V.; Wilson, John W.
- CS Sch. Phys. Sci, New Univ. Ulster, Coleraine, BT52 1SA, UK
- SO Journal of Chemical Research, Synopses (1982), (5), 132-3 CODEN: JRPSDC; ISSN: 0308-2342

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DT Journal

LA English

OS CASREACT 97:127689

IT 20631-84-9P

RL: SPN (Synthetic preparation); PREP (Preparation)

(prepn. of, by substitution reaction of bis(dimesitylboryl)methane)

RN 20631-84-9 CAPLUS

CN Borinic acid, bis(2,4,6-trimethylphenyl) - (9CI) (CA INDEX NAME)

The title compd. [(2,4,6-Me3C6H2)2B]2CH2 (I) was prepd. in 65% yield by reaction of (2,4,6-Me3C6H2)2BCH2Li with (2,4,6-Me3C6H2)2BF. Reactions of I with H2O, alcs., thiols and amines are reported. E.g., reaction of I with MeOH gave (2,4,6-Me3C6H2)2BMe and (2,4,6-Me3C6H2)2BOMe quant. Deprotonation of I with KH in THF gave the anion [(2,4,6-Me3C6H2)2B]2CH-, 13C NMR spectroscopy of which showed the neg. charge to be delocalized over the B-C-B bonds.

L4 ANSWER 201 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

AN 1982:217904 CAPLUS

DN 96:217904

TI Novel heterocyclic systems. Part 8. Novel complexes of amides and other species with a heterocyclic boron betaine

AU Fletcher, Andrew S.; Paget, Walter E.; Smith, Keith

CS Dep. Chem., Univ. Coll. Swansea, Swansea, SA2 8PP, UK

SO Heterocycles (1982), 18(Spec. Issue), 107-11

CODEN: HTCYAM; ISSN: 0385-5414

DT Journal

LA English

IT 81805-26-7P 81805-27-8P 81805-28-9P

81805-29-0P 81805-30-3P 81805-31-4P

81805-32-5P 81805-33-6P 81805-34-7P

81805-35-8P 81805-36-9P 81805-37-0P

RL: PRP (Properties); SPN (Synthetic preparation); PREP (Preparation) (prepn. and structure of)

RN 81805-26-7 CAPLUS

CN Boron, [.mu.-[[bis(1-methylethyl)amino]carbonyl-C:O]][N,N-bis(1-methylethyl)formamide-O]hydroxytetraphenyldi- (9CI) (CA INDEX NAME)

10085368.2

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RN 81805-27-8 CAPLUS

CN Boron, [.mu.-[[bis(1-methylethyl)amino]carbonyl-C:0]](N,N-dimethylformamide-O)hydroxytetraphenyldi- (9CI) (CA INDEX NAME)

RN 81805-28-9 CAPLUS

CN Boron, (acetamide-0) [.mu.-[[bis(1-methylethyl)amino]carbonyl-C:0]]hydroxytetraphenyldi- (9CI) (CA INDEX NAME)

RN 81805-29-0 CAPLUS

CN Boron, [.mu.-[[bis(1-methylethyl)amino]carbonyl-C:0]]hydroxy(N-methylacetamide-O)tetraphenyldi- (9CI) (CA INDEX NAME)

RN 81805-3.0-3 CAPLUS

CN Boron, [.mu.-[[bis(1-methylethyl)amino]carbonyl-C:0]](N,N-dimethylacetamide-O)hydroxytetraphenyldi-(9CI) (CA INDEX NAME)

RN 81805-31-4 CAPLUS

CN Boron, [.mu.-[[bis(1-methylethyl)amino]carbonyl-C:O]](cyclohexanamine)hydroxytetraphenyldi- (9CI) (CA INDEX NAME)

RN. 81805-32-5 CAPLUS

CN Boron, (benzenemethanamine) [.mu.-[[bis(1-methylethyl)amino]carbonyl-C:0]]hydroxytetraphenyldi-(9CI) (CA INDEX NAME)

RN 81805-33-6 CAPLUS

CN Boron, aqua[.mu.-[[bis(1-methylethyl)amino]carbonyl-C:O]]hydroxytetraphenyldi- (9CI) (CA INDEX NAME)

$$C = \begin{bmatrix} C & & & \\$$

RN 81805-34-7 CAPLUS

CN Boron, (benzamide-0) [.mu.-[[bis(1-methylethyl)amino]carbonyl-C:0]]hydroxytetraphenyldi- (9CI) (CA INDEX NAME)

RN 81805-35-8 CAPLUS

CN Boron, [.mu.-[[bis(1-methylethyl)amino]carbonyl-C:0]]hydroxytetraphenyl(N-phenylacetamide-O)di- (9CI) (CA INDEX NAME)

RN 81805-36-9 CAPLUS

CN Boron, [.mu.-[[bis(1-methylethyl)amino]carbonyl-C:O]](N-cyclohexylcyclohexanamine)hydroxytetraphenyldi- (9CI) (CA INDEX NAME)

RN 81805-37-0 CAPLUS

CN Boron, [.mu.-[[bis(1-methylethyl)amino]carbonyl-C:0]]hydroxy[N-(1-methylethyl)-2-propanamine]tetraphenyldi- (9CI) (CA INDEX NAME)

GI

AB The B-contg. betaine I forms stable 1:1 addn. complexes with H2O, amines and simple amides. NMR data indicate that the 1:1 complex of I with HCON(CHMe2)2 exists as II.

L4 ANSWER 202 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

AN 1982:143185 CAPLUS

DN 96:143185

TI A new method for the generation of a boron enolate of an ester. A new synthesis of 2-deoxy-D-ribose

AU Murakami, Masahiro; Mukaiyama, Teruaki

CS Fac. Sci., Univ. Tokyo, Tokyo, 113, Japan

SO Chemistry Letters (1982), (2), 241-4

CODEN: CMLTAG; ISSN: 0366-7022

DT Journal

LA English

IT 2622-89-1

RL: RCT (Reactant); RACT (Reactant or reagent)
 (reaction of, with ethoxyacetylene and aldehydes in presence of
 mercuric acetate)

RN 2622-89-1 CAPLUS

CN Borinic acid, diphenyl- (6CI, 7CI, 8CI, 9CI) (CA INDEX NAME)

AB A boron enolate of an ester was generated in situ by the treatment of EtOC.tplbond.CH with Hg(OAc)2 and Ph2BOH, and the boron enolate thus formed further reacted with aldehydes to give .beta.-hydroxyesters and .beta.-acetoxy esters in good yields. The reaction was successfully applied to the stereoselective synthesis of 2-deoxy-D-ribose.

L4 ANSWER 203 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

AN 1981:604028 CAPLUS

DN 95:204028

TI Synthesis of organoboron siloxanes and their reactions with organic hydroperoxides

AU Chistova, E. V.; Alyasov, V. N.; Galiullina, R. F.; Maslennikov, V. P.; Dodonov, V. A.; Aleksandrov, Yu. A.

CS Nauchno-Issled. Inst. Khim., Gorkiy, USSR

SO Zhurnal Obshchei Khimii (1981), 51(5), 1078-85 CODEN: ZOKHA4; ISSN: 0044-460X

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10085368.2
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DT Journal LA Russian

IT 2622-89-1

RL: RCT (Reactant); RACT (Reactant or reagent)
 (reaction of, with triethyl- and triphenylsilanes)

RN 2622-89-1 CAPLUS

CN Borinic acid, diphenyl- (6CI, 7CI, 8CI, 9CI) (CA INDEX NAME)

Ph | Ph— B— OH

AB R3SiOBR12 [R = Et, R1 = Ph (I); R = R1 = Ph] were obtained in 80 and 73% yields by treatment of Ph2BOH with R3SiH at room temp. in the presence of H2PtCl6. Treatment of Bu2BOH with Et3SiOH in refluxing C6H6 gave 70% Et3SiOBBu2. Reaction of I with R2OOH (R2 = Me3C, PhCMe2) followed by hydrolysis of the resulting reaction mixt. gave Et3SiOH, PhB(OH)2, Me3COH, MeCH(OH)Me, and PhCMe:CH2. The reaction mechanism involves a reversible intermediate exchange of R3SiO in the starting borinate with the alkylperoxy group.

L4 ANSWER 204 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

AN 1981:515504 CAPLUS

DN 95:115504

TI Dioxazaborenines

AU Moehrle, Hans; Zuege, Erika

CS Inst. Pharm. Chem., Univ. Duesseldorf, Duesseldorf, 4000/1, Fed. Rep. Ger.

SO Archiv der Pharmazie (Weinheim, Germany) (1981), 314(7), 580-7 CODEN: ARPMAS; ISSN: 0365-6233

DT Journal

LA German

IT 2622-89-1

RL: RCT (Reactant); RACT (Reactant or reagent) (reaction of, with bis(hydroxynaphthylmethyl)amine)

RN 2622-89-1 CAPLUS

CN Borinic acid, diphenyl- (6CI, 7CI, 8CI, 9CI) (CA INDEX NAME)

Ph | Ph— B— OH

GI

AB Reaction of 2-C10H7OH with hexamethylenetetramine in the presence of B(OH)3 in EtOCH2CH2OH gave the dioxazaborinane I (R = OH), which upon acid hydrolysis gave (2,1-HOC10H6CH2)2NH (II). Reaction of II with PhB(OH)2 gave I (R = Ph), but reaction with (Ph2B)2O gave the labile chelate III, which upon heating undergoes amine elimination to give IV and V.

L4 ANSWER 205 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

AN 1981:149549 CAPLUS

DN 94:149549

TI Asymmetrically substituted diphenylcarbazones as chelate formers

AU Czech, N.; Friese, B.; Umland, F.

CS Anorg. Chem. Inst., Wilhelms-Univ. Muenster, Muenster, D-4400, Fed. Rep. Ger.

SO Analytica Chimica Acta (1980), 121, 275-9 CODEN: ACACAM; ISSN: 0003-2670

DT Journal

LA English

IT 2622-89-1

RL: ANT (Analyte); ANST (Analytical study) (detn. of, (nitrophenyl)phenylcarbazones in spectrophotometric)

RN 2622-89-1 CAPLUS

CN Borinic acid, diphenyl- (6CI, 7CI, 8CI, 9CI) (CA INDEX NAME)

AB Two isomeric asym. substituted diphenylcarbazones, 1-(4-nitrophenyl)-5-phenylcarbazone and 1-phenyl-5-(4-nitrophenyl)carbazone, were synthesized by introducing 1 nitro group selectively. Their chelate complexes with several cations show increased molar absorptivities compared to those of the unsubstituted reagents, thus giving improved anal. sensitivity. The structure of the chelates is discussed; a 5-membered ring structure is very plausible.

L4 ANSWER 206 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

AN 1980:620885 CAPLUS

DN 93:220885

TI Reaction of diethylzinc with dialkyl(aryl)boric acids AU Galiullina, R. F.; Chistova, E. V.; Dodonov, V. A. CS Nauchno-Issled. Inst. Khim., Gorkiy, USSR

SO Zhurnal Obshchei Khimii (1980), 50(7), 1657-8 CODEN: ZOKHA4; ISSN: 0044-460X

DT Journal

LA Russian

IT

62981-91-3
RL: RCT (Reactant); RACT (Reactant or reagent)
 (reaction of, with diethylzinc)

RN 62981-91-3 CAPLUS

CN Borinic acid, di-1-naphthalenyl- (9CI) (CA INDEX NAME)

AB Reaction of Et2Zn with R2BOH in toluene at room temp. gave EtZnOBR2 (I, R = Bu, .alpha.-naphthyl). Hydrolysis of I with H2O gave EtH, Zn(OH)2 and R2BOH. Treating I with iodine gave EtI and R2BOZnI.

L4 ANSWER 207 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

AN 1980:586433 CAPLUS

DN 93:186433

TI Dimesitylboryl compounds. Part III. Oxygen derivatives

AU Brown, N. M. D.; Davidson, F.; McMullan, R.; Wilson, J. W.

CS Sch. Phys. Sci., New Univ. Ulster, Coleraine, UK

SO Journal of Organometallic Chemistry (1980), 193(2), 271-82 CODEN: JORCAI; ISSN: 0022-328X

DT Journal

LA English

IT 20631-84-9P

RL: PRP (Properties); SPN (Synthetic preparation); PREP (Preparation) (prepn. and NMR of)

RN 20631-84-9 CAPLUS

CN Borinic acid, bis(2,4,6-trimethylphenyl) - (9CI) (CA INDEX NAME)

AB Thirteen (2,4,6-Me3C6H2)2BOR (R = H, alkyl, CH2CH:CH2, CH2C.tplbond.CH, Ph, PhCH2, substituted-phenyl) were prepd. by treating dimesityl borinic acid with the corresponding alc. and their NMR data detd. The corresponding data for fluorodimesitylborane was also reported. As has

been found for other dimesitylboryl systems, the mesityl 13C chem. shifts remain virtually unchanged with change in R. Thus, there is no B-aryl .pi. backbonding in such systems and that B-X .pi. bonding increases in the order N>O>F>C.

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ANSWER 208 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN
L4
    1980:567026 CAPLUS
AN
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DN 93:167026

TΙ Comparison between boron-11 and carbon-13 chemical shifts of threefold coordinated boron compounds and carbenium ions

ΑU Wrackmeyer, Bernd

CS Inst. Anorg. Chem., Univ. Muenchen, Munich, D-8000, Fed. Rep. Ger.

SO Zeitschrift fuer Naturforschung, Teil B: Anorganische Chemie, Organische Chemie (1980), 35B(4), 439-46 CODEN: ZNBAD2; ISSN: 0340-5087

DT Journal

LΑ German

ΙT 2622-89-1

> RL: PRP (Properties) (NMR of boron-11 in)

2622-89-1 CAPLUS RN

Borinic acid, diphenyl- (6CI, 7CI, 8CI, 9CI) (CA INDEX NAME) CN

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Ph
Ph-B-OH
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AB The relationship between 11B chem. shifts (.delta.11B) of trigonal boranes and 13C+ chem. shifts (.delta.13C+) of carbenium ions was more complex than previously reported. The trends obsd. allow for the comparison of (pp) .pi. bonding between B and suitable substituents with the .pi.-charge delocalization in carbenium ions. In case of the ferrocenyl boranes and ferrocenyl carbenium ions the markedly different trend in the shielding of 11B and 13C+ favors a fulvene-like structure for the substituted cyclopentadienyl ring in the latter. Structural features are analogously reflected by .delta.11B and .delta.13C+ data for both series of compds. Comparison of .delta.13C data of organoboranes and carbenium ions is useful in conformational studies.

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ANSWER 209 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN
L4
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1980:214384 CAPLUS AN

DN 92:214384

TITransformation of pyrocatechols with diaryl borinic acids to paramagnetic borate complexes

ΑU Stegmann, Hartmut B.; Denninger, Guenter; Scheffler, Klaus

CS Inst. Org. Chem., Univ. Tuebingen, Tuebingen, D-7400/1, Fed. Rep. Ger.

SO Tetrahedron Letters (1979), (39), 3689-90 CODEN: TELEAY; ISSN: 0040-4039

DTJournal

LΑ German

IT 2622-89-1 66117-64-4 73774-44-4 73774-45-5

RL: RCT (Reactant); RACT (Reactant or reagent) (reaction of, with pyrocatechols)

RN 2622-89-1 CAPLUS

CNBorinic acid, diphenyl- (6CI, 7CI, 8CI, 9CI) (CA INDEX NAME)

RN 66117-64-4 CAPLUS

CN Borinic acid, bis(4-methylphenyl) - (9CI) (CA INDEX NAME)

RN 73774-44-4 CAPLUS

CN Borinic acid, bis(2-methylphenyl) - (9CI) (CA INDEX NAME)

RN 73774-45-5 CAPLUS

CN Borinic acid, bis(4-methoxyphenyl) - (9CI) (CA INDEX NAME)

GI For diagram(s), see printed CA Issue.

AB HOBR2 (R = Ph, o- and p-tolyl, p-MeOC6H4) reacted with catechols I (R1 = Ph, Me) giving stable paramagnetic complexes II (same R, R1). ESR spectra of the solns. show the H hyperfine splitting of the catechol and the coupling with the 11B and 10B nuclei.

L4 ANSWER 210 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

AN 1980:164076 CAPLUS

DN 92:164076

TI Recovery of triarylboranes

IN Seidel, William C.

PA du Pont de Nemours, E. I., and Co., USA

SO U.S., 5 pp.

CODEN: USXXAM

DT Patent

LA English

FAN.CNT 1

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	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
ΡI	US 4177215	Α	19791204	US 1978-925550	19780717
	CA 1097691	A1	19810317	CA 1978-315024	19781031
	•			US 1978-925550	19780717

IT 2622-89-1

RL: RCT (Reactant); RACT (Reactant or reagent)
 (sepn. of triphenylborane from)

RN 2622-89-1 CAPLUS

CN Borinic acid, diphenyl- (6CI, 7CI, 8CI, 9CI) (CA INDEX NAME)

Ph3B was sepd. from Ph2BOH in basic aq. soln. in which the wt. ratio of Ph3B to Ph2BOH is <13:1. The soln. was neutralized to a pH .ltoreq. 0.037 [I]2 + 0.048 [I] + 8.75 + log [Ph2BO-] where I = the ionic strength of the soln. which is maintained at 1M and [Ph2BO-] is the concn. of the salt of Ph2BOH in mol/L. Thus, an aq. soln. contg. 4.8 g Ph3B, 0.7 mL Ph2BOCHMe2, 2.8 g NaCl, and 1.2 g NaOH (wt. ratio of Ph3B-Ph2BOH = 7:9) was titrated with 1.074N HCl to a final pH of 7.6 and a borinate anion concn. of 0.043 mol. A white solid contg. 4.29 g Ph3B (96.1% yield from the adduct) and 0.152 g of Ph2BOH was recovered (Ph3B/Ph2BOH = 28). Only trace amts. of Ph3B were detected in the filtrate.

L4 ANSWER 211 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

AN 1979:525665 CAPLUS

DN 91:125665

TI Corrosion inhibitors as preservatives for metalworking fluids - ethanolamines

AU Bennett, E. O.

CS Univ. Houston, Houston, TX, 77004, USA

SO Lubrication Engineering (1979), 35(3), 137-44 CODEN: LUENAG; ISSN: 0024-7154

DT Journal

LA English

IT 71173-48-3

RL: USES (Uses)

(lubricating oil additive, as corrosion inhibitor and antimicrobial agent)

RN 71173-48-3 CAPLUS

CN Borinic acid, diphenyl-, compd. with 2-aminoethanol (1:1) (9CI) (CA INDEX NAME)

CM 1

CRN 2622-89-1 CMF C12 H11 B O

9/24/2003>

CM 2

CRN 141-43-5 CMF C2 H7 N O

 $H_2N - CH_2 - CH_2 - OH$

AB Fifty-nine monoethanolamines, diethanolamines, and triethanolamines were studied for their antimicrobial properties in 13 cutting fluid products. 2-(N-Amyl) ethanolamine [35161-67-2] exhibited outstanding activity in all of the products. Other compds. producing significant inhibition of microbial growth included N-Me ethanolamine [109-83-1], N-Et ethanolamine [110-73-6], N-Bu ethanolamine [111-75-1], 2-N-methyl-N-heptyl) ethanolamine [71247-70-6], 2-cyclohexyl ethanolamine [2842-38-8], and N-benzyl ethanolamine [104-63-2].

L4 ANSWER 212 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

AN 1979:187016 CAPLUS

DN 90:187016

TI Thermal decomposition of organoborate salts

AU Fields, C. L.; Patnoe, R. L.; Leschnik, D.

CS Dep. Chem., Univ. Northern Colorado, Greeley, CO, USA

SO Analytical Calorimetry (1977), 4, 91-3

CODEN: ANCAD4; ISSN: 0066-1538

DT Journal

LA English

IT 70149-20-1 70149-22-3 70149-24-5

RL: RCT (Reactant); RACT (Reactant or reagent)
 (thermal decompn. of)

RN 70149-20-1 CAPLUS

CN Borate(1-), dihydroxydiphenyl-, lithium, dihydrate, (T-4)- (9CI) (CA INDEX NAME)

● Li+

●2 H₂O

RN 70149-22-3 CAPLUS

CN Borate(1-), dihydroxydiphenyl-, (T-4)-, sodium, compd. with 2-propanol (1:1) (9CI) (CA INDEX NAME)

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10085368.2
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CM1

CRN 70149-21-2

CMF C12 H12 B O2 . Na

CCI CCS

Na +

CM 2

CRN 67-63-0

CMF C3 H8 O

RN

70149-24-5 CAPLUS
Borate(1-), hydroxytriphenyl-, (T-4)-, lithium, compd. with CN1,1'-oxybis[ethane] (1:1) (9CI) (CA INDEX NAME)

CM1

CRN 70149-23-4

CMF C18 H16 B O . Li

CCI CCS

Li+

CM2

CRN 60-29-7 CMF C4 H10 O

H3C-CH2-O-CH2-CH3

Thermal decompn. of M[PhB(OH)3] (I) and M[Ph2B(OH)2] Q [M = Li, Na; Q = AB 2H2O, Me2CHOH] gave benzene and MBO2. Decompn. of I proceeded through 1 or more intermediate phases.

ANSWER 213 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN L4

AN 1978:559644 CAPLUS

DN 89:159644

Effects of phenylboronic and diphenylborinic acid on the paper TIchromatographic mobilities of cardenolides and bufadienolides

ΑU Megges, R.; Streckenbach, Barbara; Repke, K. R. H.

Zentralinst. Molekularbiol., DAW, Berlin, Ger. Dem. Rep. CS

SO Journal of Chromatography (1978), 155(1), 169-77 CODEN: JOCRAM; ISSN: 0021-9673

DT

Journal

German LΑ

ΙT 2622-89-1

RL: ANST (Analytical study)

(bufadienolides and cardenolides paper chromatog, in presence of)

RN 2622-89-1 CAPLUS

CN Borinic acid, diphenyl- (6CI, 7CI, 8CI, 9CI) (CA INDEX NAME)

Ph Ph-B-OH

AΒ Phenylboronic acid and diphenylborinic acid were studied as to their influence on the paper chromatog. mobilities of cardenolides and bufadienolides with and without a cis-1,2- or cis-1,3-diol group. At low concns., phenylboronic acid and, at higher concns., diphenylborinic acid increased the mobility of nearly all investigated cis-1,3-diols with a tertiary OH-group. At higher concns. both acids enhanced the mobilities of most of the cis-1,3- and cis-1,2-diols without a tertiary OH-group. Thus, there are 2 basic prerequisites for the derivatization of cis-1,2or cis-1,3-diols: (1) the capabilities of the diol to reach an O-O distance like that in phenylboronic acid esters or in diphenylborinic acid complexes and, (2) the absence of a considerable steric hindrance by a substituent near the reactive diol group. Among the 4 rhamnosides studied, being 1,2-diols, 2 do not react for unknown reasons.

- L4ANSWER 214 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN
- AN 1978:546345 CAPLUS
- 89:146345 DN
- ΤI New method for catalytic synthesis of unsaturated alcohols from butadiene and boric acid
- ΑU Džhemilev, U. M.; Kunakova, R. V.; Minsker, D. L.; Vasil'eva, E. V.; Tolstikov, G. A.
- CS Inst. Khim., Ufa, USSR

SO Izvestiya Akademii Nauk SSSR, Seriya Khimicheskaya (1978), (6), 1466-7 CODEN: IASKA6; ISSN: 0002-3353 DT Journal

LA Russian IT 2622-89-1

RL: RCT (Reactant); RACT (Reactant or reagent) (telomerization of butadiene with, unsatd. alcs. by)

RN 2622-89-1 CAPLUS

CN Borinic acid, diphenyl- (6CI, 7CI, 8CI, 9CI) (CA INDEX NAME)

Ph | Ph-- B-- OH

AB Treating butadiene with 20% aq. H3BO3 in 6:1 ratio in PhMe contg. 1:3:4 Pd(acac)2-Ph3P-Et3Al for 10 h at 70.degree. gave 25:15:60 CH2:CH(CH2)3CH:CHCH2OH (I)-CH2:CH(CH2)3CH(OH)CH:CH2 (II)-CH2:CHCH2(CH2CH:CHCH2)2OH in 80% combined yield after hydrolysis. Using Ph2BOH for H3BO3 gave only I and II via the intermediate diphenylborinate esters.

L4 ANSWER 215 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

AN 1978:136709 CAPLUS

DN 88:136709

TI Reactions of diborane with organic derivatives of tin and lead

AU Thorpe, F. G.; Breuer, S. W.; Pickles, G. M.; Spencer, T.; Podesta, J. C.

CS Chem. Dep., Univ. Lancaster, Lancaster, UK

SO Journal of Organometallic Chemistry (1978), 145(3), C26-C28 CODEN: JORCAI; ISSN: 0022-328X

DT Journal

LA English

IT 2622-89-1P 66117-64-4P

RL: FORM (Formation, nonpreparative); PREP (Preparation) (formation of, from hydrolysis of intermediates from reaction of diborane with phenyltin and phenyllead compds.)

RN 2622-89-1 CAPLUS

CN Borinic acid, diphenyl- (6CI, 7CI, 8CI, 9CI) (CA INDEX NAME)

Ph | Ph-- B-- OH

RN 66117-64-4 CAPLUS

CN Borinic acid, bis(4-methylphenyl)- (9CI) (CA INDEX NAME)

AB Diborane reacts with aryltin (e.g. Ph4Sn) and aryllead compds. to give intermediates which on hydrolysis give arylboronic (e.g. PhB(OH)2) and arylborinic acids, and on oxidn. give phenols (e.g. PhOH).

L4 ANSWER 216 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

AN 1978:39662 CAPLUS

DN 88:39662

TI Catalytic detoxification of combustion waste gases

IN Brantl, Victor

PA BRASEC G.m.b.H. Chemisch-Physikalisches Laboratorium, Fed. Rep. Ger.

SO Ger., 4 pp. CODEN: GWXXAW

DT Patent

LA German

FAN.CNT 1

PATENT NO. KIND DATE APPLICATION NO. DATE

PI DE 2500683 A1 19760715 DE 1975-2500683 19750109

DE 1975-2500683 19750109

IT 2622-89-1

RL: USES (Uses)

(gasoline additives, for exhaust gas pollutant redn.)

RN 2622-89-1 CAPLUS

CN Borinic acid, diphenyl- (6CI, 7CI, 8CI, 9CI) (CA INDEX NAME)

Ph | Ph— B— OH

- AB Defined amts. of certain Si, Al, B, Os, Pt, Ge, Sb, Sn, Ti, Pb, Cu, Zn, or Cr compds. are added to fuels either alone or in mixts., to reduce NO to N and to oxidize CO to CO2 during combustion. The fuels (e.g., gasoline) are sprayed into the combustion chamber during the combustion step. The amts. of the compds. added are given in tabular form.
- L4 ANSWER 217 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN
- AN 1977:406060 CAPLUS
- DN 87:6060
- TI Naphthylboryne: a monovalent organoboron carbene analog from photolysis of tri-1-naphthylboron
- AU Ramsey, Brian G.; Anjo, Dennis M.
- CS Dep. Chem., San Francisco State Univ., San Francisco, CA, USA
- SO Journal of the American Chemical Society (1977), 99(9), 3182-3 CODEN: JACSAT; ISSN: 0002-7863
- DT Journal
- LA English
- IT 62981-91-3P

RN 62981-91-3 CAPLUS

CN Borinic acid, di-1-naphthalenyl- (9CI) (CA INDEX NAME)

Patel 9/24/2003>

AB Cyclohexanol and cis-1,2-cyclohexanediol are isolated after oxidn. and hydrolysis of the products of tri-1-naphthylboron (I) photolysis in cyclohexane and cyclohexene resp. Their isolation is evidence of C-H insertion and addn. to olefin bonds by the reactive intermediate, naphthylboryne (II). Products indicative of carbon chlorine insertion are obtained from photolysis of I in CCl4. II is formed by a di-pi.-methane mechanism from I.

L4 ANSWER 218 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

AN 1976:560214 CAPLUS

DN 85:160214

TI Boron-nitrogen compounds. LXI. Studies on (2-pyridylamino)diphenylborane and some related species

AU Gragg, B. R.; Niedenzu, K.

CS Dep. Chem., Univ. Kentucky, Lexington, KY, USA

SO Journal of Organometallic Chemistry (1976), 117(1), 1-11 CODEN: JORCAI; ISSN: 0022-328X

DT Journal

LA English

IT 61226-22-0P

RL: SPN (Synthetic preparation); PREP (Preparation) (prepn. of)

RN 61226-22-0 CAPLUS

CN Boron, hydroxytetraphenyl[.mu.-(2-pyridinaminato-N1:N2)](2-pyridinamine-N1)di- (9CI) (CA INDEX NAME)

GΙ

AB (2-Aminopyridine) triphenylborane was prepd. from its acid-base pair constituents; pyrolysis of which yields borazine I (R = 2-pyridyl). An intermediate in the transamination of Me2NBPh2 with 2-aminopyridine to yield (2-pyridylamino) diphenylborane was isolated and was the 1:1 molar adduct II of Me2NBPh2 with 2-aminopyridine. Related to this 1:1 adduct is the initial product of the interaction of (2-pyridylamino) diphenylborane with water, which is the 1:1:1 adduct III of (2-pyridylamino) diphenylborane, HOBPh2 and 2-aminopyridine; pyrolysis of III yields IV. IV was also obtained from the interaction of (2-pyridylamino) diphenylborane with 0 and with AcPh via 0 abstraction from the latter. (2-Pyridylamino) diphenylborane 1,2-aminoboronates one CO double bond of CO2.

L4 ANSWER 219 OF 309 CAPLUS. COPYRIGHT 2003 ACS on STN

AN 1975:131728 CAPLUS

DN 82:131728

TI Correlations between carbon-13 and boron-11 chemical shifts. IV. Carbenium ions and their trigonal boron analogs

AU Spielvogel, Bernard F.; Nutt, W. Rodger; Izydore, Robert A.

CS Paul M. Gross Chem. Lab., Duke Univ., Durham, NC, USA

SO Journal of the American Chemical Society (1975), 97(6), 1609-10 CODEN: JACSAT; ISSN: 0002-7863

DT Journal

LA English

IT 2622-89-1

RL: PRP (Properties)

(NMR of, correlation with carbon-13 chem. shifts in carbenium ion analogs)

RN 2622-89-1 CAPLUS

CN Borinic acid, diphenyl- (6CI, 7CI, 8CI, 9CI) (CA INDEX NAME)

AB The 13C chem. shifts of 15 carbenium ions were compared with the 11B

9/24/2003>

Patel

shifts in the analogous trigonal boron compds. The 11B shifts for 4 compds. were calcd. by using a pairwise additivity rule. With the exception of the triphenyl and cyclopropyldimethyl derivs., the shifts are linearly related. Least squares anal. of the 1st 9 compds. yields the equation, .delta.(11B) = $0.384.\mathrm{sigma.}(13C)$ - 31.6. The slope is very close to that previously reported (0.40) in the linear correlation of 13C and 11B chem. shifts data between tetracoordinate boron and carbon species. The 13C shifts for a series of diphenyl- and mono-phenylcarbenium ions are pairwise additive, but with inclusion of the triphenylcarbenium ion, the rule is n longer obeyed. Discussions concerning the cyclopropyldimethyl and triphenyl derivs. are presented.

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L4 ANSWER 220 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN
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AN 1975:104683 CAPLUS

DN 82:104683

TI Reactor reactivity control by coolant passage coating

IN Wheelock, Clifford W.

PA United States Atomic Energy Commission

SO U.S., 8 pp. CODEN: USXXAM

DT Patent

LA English

FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
ΡI	US 3860482	Α	19750114	US 1957-686262	19570925
				US 1957-686262	19570925

IT 2622-89-1

RL: PROC (Process)

(coatings, in nuclear reactor coolant channels, for reactivity control)

RN 2622-89-1 CAPLUS

CN Borinic acid, diphenyl- (6CI, 7CI, 8CI, 9CI) (CA INDEX NAME)

AB A method is described for controlling the reactivity in a nuclear reactor cooled by water. A n absorbing soln., consisting of a Cd compd., together with HF, is injected at a predetd. flux level into the coolant water. At least part of the Cd is deposited on the interior walls (of stainless steel or Al) of the cooling channels, thereby reducing the n multiplication factor of the reactor. For example, a n poison material consisting of a soln. of 50 wt. % Cd octoate [2191-10-8] in a compn. consisting of PhOH 37.5, resin of the glycerol ester of maleic acid 15, resin of the Me ester of abietic acid 45, and Et cellulose 2.5 parts is contained in a storage chamber attached to the cooling coil on the outside of the reactor core tank. Ar under pressure is used to force the soln. into the cooling channel when the n flux reaches 3 .times. 1012 thermal n/cm2-sec. About 6 of the 8 g of Cd in the soln. is deposited on the internal surface of the cooling tube within the reactor core. The n multiplication factor is reduced by .apprx.3%. The poison is removed by trichloroethylene.

L4 ANSWER 221 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN AN 1974:83113 CAPLUS

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10085368.2
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Page 301

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DN
     80:83113
TI
     Heterocyclic organoboron compounds. XVI. Chelated compounds with
     .alpha.,.beta.-unsaturated .beta.-amino ketones
AU
     Bally, I.; Ciornei, E.; Vasilescu, A.; Balaban, A. T.
CS
     Inst. At. Phys., Bucharest, Rom.
SO
     Tetrahedron (1973), 29(20), 3185-7
     CODEN: TETRAB; ISSN: 0040-4020
DT
     Journal
     English
LΑ
ΙT
     2622-89-1
     RL: RCT (Reactant); RACT (Reactant or reagent)
        (reaction with aminobenzylideneacetophenone)
     2622-89-1 CAPLUS
RN
CN
     Borinic acid, diphenyl- (6CI, 7CI, 8CI, 9CI) (CA INDEX NAME)
   Ph
Ph-B-OH
GI
     For diagram(s), see printed CA Issue.
AΒ
     .alpha.,.beta.-Unsatd. .beta.-amino ketones reacted with B compds. (e.g.
     Ph2BOH, BF3) to give chelates. Thus Ph2BOH with MeCOCH: CMeNH2 gave I.
    ANSWER 222 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN
T.4
AN
     1974:47322 CAPLUS
     80:47322
DN
TI
     Determination of the hydroxyquinolizidine configuration in Sabadilla
     alkamines with boron complexes
    Moehrle, H.; Clauss, E.
ΑU
CS
     Pharm. Inst., Freie Univ. Berlin, Berlin, Fed. Rep. Ger.
SO
    Archiv der Pharmazie (Weinheim, Germany) (1973), 306(10), 721-9
     CODEN: ARPMAS; ISSN: 0365-6233
DT
     Journal
LΑ
    German
IT
     51210-41-4P 51210-42-5P
    RL: SPN (Synthetic preparation); PREP (Preparation)
        (prepn. of)
     51210-41-4 CAPLUS
RN
CN
     Cevane-3,4,12,14,16,17,20-heptol, 4,9-epoxy-, (3.alpha.,4.alpha.,16.beta.)-
     , diphenylborinate (salt) (9CI) (CA INDEX NAME)
     CM
     CRN
          2622-89-1
     CMF
         C12 H11 B O
   Ph
Ph-B-OH
     CM
          2
    CRN
         124-98-1
```

CMF C27 H43 N O8

Absolute stereochemistry.

RN 51210-42-5 CAPLUS
CN Cevane-3,4,12,14,16,17,20-heptol, 4,9-epoxy-, (3.beta.,4.alpha.,16.beta.), diphenylborinate (salt) (9CI) (CA INDEX NAME)

CM 1

CRN 5876-23-3 CMF C27 H43 N O8

Absolute stereochemistry.

CM 2

CRN 2622-89-1 CMF C12 H11 B O

GI For diagram(s), see printed CA Issue.

AB Hydroxy-substituted amines as model compds., e.g., I (n = 0), reacted with pure Ph2BOH, freshly prepd. by HClO4-treatment of the boroxazolidine of 1-phenyl-2-morpholinoethanol and Na tetraphenylborate, to give the corresponding salts, e.g., II, whereas the analogous N-oxides I (n = 1) gave cyclic B betaines, e.g., III. Similarly reacted Sabadilla alkamines of quinolizidine type, e.g. cevine or veracevine, indicating the retention of the configuration during the prepn. of the N-oxides and the original stereochem. of the alkamines.

L4 ANSWER 223 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

AN 1974:14452 CAPLUS

DN 80:14452

TI Conformational dynamics of alkoxydiarylboranes

AU Finocchiaro, Paolo; Gust, Devens; Mislow, Kurt

CS Dep. Chem., Princeton Univ., Princeton, NJ, USA

SO Journal of the American Chemical Society (1973), 95(21), 7029-36 CODEN: JACSAT; ISSN: 0002-7863

DT Journal

LA English

IT 50481-12-4P

RL: SPN (Synthetic preparation); PREP (Preparation)

(prepn. of)
RN 50481-12-4 CAPLUS

CN Borinic acid, bis(4-methoxy-2,6-dimethylphenyl)- (9CI) (CA INDEX NAME)

AB The low-temp. 1H-NMR spectra of several alkoxydiarylboranes are shown to be consistent with three alternatives: the mols. adopt propeller conformations, and stereoisomerization is slow on the NMR time scale; the mols. adopt propeller conformations with rapid stereoisomerization by means of one-flip pathways; the molecules adopt perpendicular conformations with slow stereoisomerization. Stereoisomerization phenomena obsd. at elevated temps. are discussed in terms of flip mechanisms, and pathways which involve the flipping of aryl groups only are found to have lower activation energies than those which also involve flipping of the alkoxy group. An anal. of the isomerization processes reveals restricted rotation about the B-O bond, and arguments are presented which suggest that conjugative effects between B and O play a significant role in the stereochemistry of alkoxyarylboranes.

L4 ANSWER 224 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

AN 1973:84899 CAPLUS

DN 78:84899

TI Electrolytically controlled termination of living anionic polymerization

AU Bhadani, S. N.

CS Dep. Chem., Ranchi Univ., Ranchi, India

SO Transactions of the SAEST (1972), 7(3), 98-99 CODEN: TSETA6; ISSN: 0036-0678

DT Journal

LA English

IT 2622-89-1P

RL: FORM (Formation, nonpreparative); PREP (Preparation) (formation of, in electrochemical polymn. of methylstyrene in presence of sodium tetraphenylborate)

RN 2622-89-1 CAPLUS

CN Borinic acid, diphenyl- (6CI, 7CI, 8CI, 9CI) (CA INDEX NAME)

AB Analysis of the THF anode soln. in the Na tetraphenylboron [143-66-8]-catalyzed .alpha.-methylstyrene [98-83-9] electropolymn. showed the presence of diphenylboronous acid [2622-89-1], phenol [108-95-2], and biphenyl [92-52-4] as terminating species and indicated the occurrence of a 1-electron mechanism.

L4 ANSWER 225 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN AN 1973:20869 CAPLUS

DN 78:20869 TICritical equivalent electrical conductivities of ions in organic solvents at 25.deg. ΑU Krumgal'z, B. S. CS Sev.-Zapadn. Zaochn. Politekh. Inst., Leningrad, USSR SO Elektrokhimiya (1972), 8(9), 1320-5 CODEN: ELKKAX; ISSN: 0424-8570 DTJournal LΑ Russian ΙT 40905-43-9 RL: PRP (Properties) (elec. cond. of, in ethylene chloride) RN 40905-43-9 CAPLUS CN Borate(1-), hydroxytriphenyl-, (T-4)- (9CI) (CA INDEX NAME)

The limiting equiv. conductances of more than 70 ions in 12 nonaq. solvents were evaluated by a special method. The solvents were: formamide, DMF, dimethylacetamide, nitromethane, nitrobenzene, o-dichlorobenzene, ethylidenechloride, pyridine, adiponitrile, 1,3-diaminopropane, and formic acid. The exptl. and evaluated data of the limiting equiv. conductances of salts in various solvents at 25.degree. were compared and a much better agreement was obtained. There are no anomalies in the solvation of cations in org. solvents. For anions, the character of the solvation effects is more complicated; not only is ionic size important but the capability of forming donor-acceptor bonds with the solvent mol. must also be considered.

L4 ANSWER 226 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

AN 1971:541072 CAPLUS

DN 75:141072

TI Paper electrophoretic and NMR studies of complexes between polyols and diphenylborinic acid

AU Garegg, Per J.; Lindstrom, Krister

CS Inst. Org. Kemi, Univ. Stockholm, Stockholm, Swed.

SO Acta Chemica Scandinavica (1947-1973) (1971), 25(5), 1559-66 CODEN: ACSAA4; ISSN: 0001-5393

DT Journal

LA English

IT 2622-89-1

RL: RCT (Reactant); RACT (Reactant or reagent) (electrophoresis of carbohydrates in solns. of)

RN 2622-89-1 CAPLUS

CN Borinic acid, diphenyl- (6CI, 7CI, 8CI, 9CI) (CA INDEX NAME)

AB The electrophoretic mobilities of various aldoses, ketoses, xylose and glucose monomethyl ethers, glycosides, alditols, and inositols in diphenylborinate at pH 10 were examd. and compared with those in borate. Whereas borate can form different types of cyclic complexes with polyols, only one such complex is possible with diphenylborinate. Nevertheless, the mobilities for the various polyols in this buffer closely parallel those obtained in borate. The NMR spectrum of epi-inositol in borate at pH 10 shows inversion of the cyclohexane ring into the conformationally less stable chair form, presumably in order to form the favored tridentate complex involving 3 cis-axial oxygens and boron. By contrast, no such inversion occurs in the presence of diphenylborinate, although strong complexing (nontridentate) is obsd. In aq. solns. and under favorable steric conditions, strong tridentate complexes are formed between borate and polyols.

L4 ANSWER 227 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

AN 1970:531065 CAPLUS

DN 73:131065

TI Organoboron compounds. IX. Diborinic acids and their derivatives

AU Coutts, I. G. C.; Musgrave, Oliver C.

CS Chem. Dep., Univ. Aberdeen, Old Aberdeen, UK

SO Journal of the Chemical Society [Section] C: Organic (1970), (16), 2225-7 CODEN: JSOOAX; ISSN: 0022-4952

DT Journal

LA English

IT 29137-54-0P 29137-55-1P 29137-57-3P

29137-58-4P

RL: SPN (Synthetic preparation); PREP (Preparation)
 (prepn. of)

RN 29137-54-0 CAPLUS

CN Borinic acid, p-phenylenebis[(p-methoxyphenyl)- (8CI) (CA INDEX NAME)

RN 29137-55-1 CAPLUS

CN Borinic acid, p-phenylenebis[(p-methoxyphenyl)-, compd. with 4-(dimethylamino)pyridine (1:2) (8CI) (CA INDEX NAME)

CM 1

CRN 29137-54-0 CMF C20 H20 B2 O4

CM 2

CRN 1122-58-3 CMF C7 H10 N2



RN 29137-57-3 CAPLUS

CN Borinic acid, p-phenylenebis[phenyl- (8CI) (CA INDEX NAME)

RN 29137-58-4 CAPLUS

CN Borinic acid, 4,4'-biphenylylenebis[phenyl- (8CI) (CA INDEX NAME)

AB By treatment with Grignard reagents esters of (HO)2BRB(OH)2 are converted into RB(OH)R'B(OH)R (R = Ph, Bu, p-MeOC6H4, cyclohexyl; R1 = p-C6H4; thien-2,5-diyl, (CH2)4, (CH2)10, p-C6H4C6H4-p) which are isolated in the form of their bis-2-aminoethyl esters. Acidification of the latter enables some of the free diborinic acids to be obtained. The hydrolysis, oxidn., and dehydration of the diborinic acids are described.

L4 ANSWER 228 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

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AN
     1969:53490 CAPLUS
DN
     70:53490
ΤI
     Investigations on 1:1 chelates of curcumin with boric acid and phenylboric
ΑU
     Umland, F.; Pottkamp, F.
CS
     Univ. Muenster, Muenster, Fed. Rep. Ger.
SO
     Fresenius' Zeitschrift fuer Analytische Chemie (1968), 241(3), 223-34
     CODEN: ZACFAU; ISSN: 0016-1152
DT
     Journal
LΑ
     German
IT
     2622-89-1
     RL: RCT (Reactant); RACT (Reactant or reagent)
         (reaction of, with curcumin)
RN
     2622-89-1 CAPLUS
     Borinic acid, diphenyl- (6CI, 7CI, 8CI, 9CI) (CA INDEX NAME)
CN
   Ph
Ph-B-OH
AΒ
     Spectral studies of curcumin (I) and protonated I (10 ml. 2.7 .times.
     10-4M) I soln. in peroxide-free dioxane with 3 ml. 1:1 phenol (II) -C6H6
     and 0, 2, 4, 6, 10, or 20 ml H2SO4-HOAc) indicated that II stabilized the
     protonated I (the quinonoid form). The absorptivity for II-contg. solns.
     was 73,600 mole/1.cm. The existence of 1:1 complexes of boric acid (III), phenylboric acid (IV), and diphenylboric acid (V) with I was shown. The dissocn. consts. KD of III-I chelates were 4 .times. 10-5 and 3 .times.
     10-4 in HOAc and dioxane, resp., while KD for the IV-I complexes was 10-3
     in dioxane and 10-4 when stabilized with II. KD for the V-I complex in
     dioxane was 7 .times. 10-5. The compds. with III and IV exist as
     diacetato- and phenylacetato-chelates of I, resp.
L4
     ANSWER 229 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN
AN
     1968:447839 CAPLUS
DN
     69:47839
     Spectroscopic studies of organoboron compounds. II. Ultraviolet spectra
ΤI
     of organoboron compounds
ΑU
     Lapkin, I. I.; Yuzhakova, G. A.
CS
     USSR
SO
     Uchenye Zapiski - Permskii Gosudarstvennyi Universitet imeni A. M.
     Gor'kogo (1966), No. 159, 270-5
     CODEN: UPGGAZ; ISSN: 0372-4514
DT
     Journal
ΤιA
     Russian
ΙT
     20631-84-9
     RL: PRP (Properties)
         (spectrum (uv) of)
```

Borinic acid, bis(2,4,6-trimethylphenyl) - (9CI) (CA INDEX NAME)

RN

CN

20631-84-9 CAPLUS

AB The uv spectra (220-400 m.mu.) of alc. solns. of 22 tris(2-alkoxy-1-naphthyl)boranes and of complexes of tris(0-alkoxyphenyl)boranes with NH3, PhNH2, Me2NH, p-toluidine, and pyridine were measured. The frequencies and absorptivities of the bands are tabulated. The spectra of the above complexes are given. The changes in spectra owing to the interaction of aromatic rings of the substituents with the central B atom are discussed.

L4 ANSWER 230 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

AN 1968:447731 CAPLUS

DN 69:47731

TI Spectroscopic studies of organoboron compounds. I. Infrared spectra of organoboron compounds

AU Lapkin, I. I.; Yuzhakova, G. A.

CS USSR

SO Uchenye Zapiski - Permskii Gosudarstvennyi Universitet imeni A. M. Gor'kogo (1966), No. 159, 264-9
CODEN: UPGGAZ; ISSN: 0372-4514

DT Journal

LA Russian

IT 20631-84-9

RL: PRP (Properties)
 (spectrum (ir) of)

RN 20631-84-9 CAPLUS

CN Borinic acid, bis(2,4,6-trimethylphenyl) - (9CI) (CA INDEX NAME)

The ir spectra (750-1750 and 2750-3850 cm.-1, measured in Nujol mull) are given of 20 tris(2-alkoxy-1-naphthyl)boron compds. and of the complexes of tris(o-alkoxyphenyl)boron (I) compds. with NH3, PhNH2, p-toluidine (II), Me2NH, and pyridine. The changes in spectra due to the interaction of aromatic rings of the substituents with the central B atoms are discussed. The main bands corresponding to the aromatic ring do not appear, practically, in the spectra of I, except the line at 1600 cm.-1; only in the spectra of complexes with II, there appears clearly the 3rd of main bands at 1508-1517 cm.-1

L4 ANSWER 231 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

AN 1968:68342 CAPLUS

DN 68:68342

TI Standard enthalpy of formation of diphenylborinic acid

AU Finch, Arthur; Gardner, Peter J.; Watts, G. B.

CS Roy. Holloway Coll., Englefield Green, UK

9/24/2003>

Patel

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10085368.2
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Page 310

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SO Chemical Communications (London) (1967), (20), 1054-5
    CODEN: CCOMA8; ISSN: 0009-241X

DT Journal

LA English

IT 2622-89-1

RL: PRP (Properties)
    (heat of formation of)

RN 2622-89-1 CAPLUS

CN Borinic acid, diphenyl- (6CI, 7CI, 8CI, 9CI) (CA INDEX NAME)
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Ph | Ph— B— OH

The standard enthalpy of formation (.DELTA.Hf0) of the title compd. (I) was detd. using standard ancillary data and data obtained from the hydrolysis of Ph2BCl in satd. aq. soln. of I and from oxidative hydrolysis of Ph2BCl in a soln. 0.1M in H2O2 and NaOH. Values for .DELTA.Hf0 of -77.4 .+-. 1.8 kcal./mole and -31.9 .+-. 1.8 kcal./mole were derived for I and Ph2BCl, resp. Using a previously obtained data for the heat of reaction of Ph2BCl, a .DELTA.Hf0 value of -16.1 .+-. 1.9 kcal./mole was obtained for Ph2BBr. By using previously known heats of vaporization for the 2 halides, the bond energies of the B-C bond in Ph2BBr and Ph2BCl were calcd. to be 109.1 .+-. 2.4 kcal./mole and 111.3 .+-. 2.4 kcal./mole, resp.

L4 ANSWER 232 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

AN 1967:32478 CAPLUS

DN 66:32478

TI Thermochemistry of phenylboronic acid, diphenylborinic acid, and their anhydrides

AU Finch, Arthur; Gardner, Peter J.

CS Roy. Holloway Coll., Englefield Green, UK

SO Transactions of the Faraday Society (1966), 62(12), 3314-18 CODEN: TFSOA4; ISSN: 0014-7672

DT Journal

LA English

IT 2622-89-1

RL: PRP (Properties)
(heat of formation of)

RN 2622-89-1 CAPLUS

CN Borinic acid, diphenyl- (6CI, 7CI, 8CI, 9CI) (CA INDEX NAME)

Ph | Ph— B— OH

AB Values for the standard enthalpies of formation of phenylboronic acid, diphenylborinic acid, phenylboronic anhydride, and phenylborninic anhydride are reported. 22 references.

L4 ANSWER 233 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

AN 1966:476761 CAPLUS

DN 65:76761

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OREF 65:14361h,14362a-b
     Seed protection with triarylborane complexes
ΙN
     Birnbaum, Herman A.; Anderson, Harvey L.
     Minnesota Mining and Manufg. Co.
PΑ
SO
     6 pp.
DT
     Patent
LΑ
     Unavailable
FAN.CNT 1
                                           APPLICATION NO.
     PATENT NO.
                      KIND DATE
                                                             DATE
                      _ _ _ _
                            ------
PΙ
     US 3268401
                            19660823
                                           US
                                                             19640615
IT
     12113-07-4, Sodium hydroxide, compd. with Ph3B (1:1)
        (mixt. with buffering agent, fungicidal activity of)
RN
     12113-07-4 CAPLUS
     Borate(1-), hydroxytriphenyl-, sodium, (T-4)- (9CI) (CA INDEX NAME)
CN
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$$\begin{array}{c|c} C & B \\ \hline \end{array}$$

• Na+

AΒ cf. CA 58, 2797d. The phytotoxicity of the title compds. (I) can be reduced by formulation with 3-10 parts of a solid buffer, e.g., Na2CO3, KHCO3, or Ca(OH)2, to maintain a pH of 7.5-12.5. Thus, the following I were prepd. by mixing, in Et2O, equimolar amts. of triarylborane and a Lewis base (II) with a pKb <10 (aryl, II, m.p.): Ph, NH3, 179-83.degree.; Ph, MeNH2, 195-213.degree.; Ph, triethylenetetramine, 75-84.degree.; Ph, Me2NH, 157-66.degree.; Ph, piperazine, 170-5.degree.; Ph, Me3N, 132-8.degree.; Ph, pyridine, 182-202.degree.; Ph, 3,5-dichloropyridine, 112-15.degree.; Ph, bis(4-pyridyl)ethylene glycol, 173-81.degree.; Ph, .gamma.-picoline, 135-45.degree.; Ph, imidazole, 185-90.degree.; Ph, NaOH, >300.degree.; .alpha.-naphthyl, NH3, 153-7.degree.; .alpha.-naphthyl, Et2NH, 170-5.degree.; .alpha.-naphthyl, Me3N, 156-8.degree.; 4-FC6H4, NH3, 179-81.degree.; 4-FC6H4, Et3N, 110-15.degree.; 4-MeOC6H4, NH3, 138-42.degree.; 4-tolyl, NH3, 143-56.degree.. Application of 0.03-1.0 oz. I/100 lb. corn, bean, oat, flax, or other seed protects the seeds from soil organisms, e.g., Pythium.

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L4 ANSWER 234 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN
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AN 1966:455961 CAPLUS

DN 65:55961

OREF 65:10403h,10404a-b

TI Hydrocarbon oils

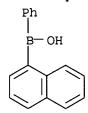
IN Timmons, Peter S.

PA "Shell" Research Ltd.

SO 9 pp.

DT Patent

LΑ Unavailable FAN.CNT 1 APPLICATION NO. DATE PATENT NO. KIND DATE _____ ----------GB 19660602 PΤ GB 1031533 19620727 13331-25-4, Borinic acid, 1-naphthylphenyl-IT (as sludge-inhibitor for fuels) RN 13331-25-4 CAPLUS CN Borinic acid, 1-naphthalenylphenyl- (9CI) (CA INDEX NAME)



AΒ Distillate hydrocarbon oils, particularly aviation kerosine, b. 80-350.degree.C. and contg. <0.01% ash to which are added 10-500 ppm. of an organoboronic or -borinic acid, e.g. phenylboronic acid, 4-chlorophenylboronic acid, 4-fluorophenylboronic acid, 4-bromophenylboronic acid, cyclohexylboronic acid, 2-furylboronic acid, 2-thienylboronic acid, and phenyl-1-naphthylborinic acid will show min. or no sludge formation or solids deposits. Numerous samples having 2 base oils (aviation kerosine) contg. various amts. of additives were subjected to 2 tests: (1) a modified form of CFR Fuel Coker Test (ASTM D-1660), in which a tank temp. of 300.degree.F., a preheater temp. of 350.degree.F., and a filter temp. of 450.degree.F. were used in place of the lower specified temps.; and (2) a bottle stability test, which consists in heating 100-ml. samples in 150-ml. conical flasks at 140.degree.C. for 14 days. A fuel which deposits sludge or is darker than a yellow color for up to 7 days fails to pass the test; the sludge is assessed visually after 14 days. The data showed that the compns. contg. the organoboron compd. gave less solid deposits and improved CFR Fuel Coker tests than the distillate hydrocarbon oils alone.

L4ANSWER 235 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN AN . 1966:443852 CAPLUS DN 65:43852 OREF 65:8186f-g TIElectric dipole hyperpolarizabilities for S-state atoms and ions ΑU Langhoff, P. W.; Lyons, J. D.; Hurst, R. P. Cornell Aeron. Lab., Buffalo, NY CS SO Physical Review (1966), 148(1), 18-25 CODEN: PHRVAO; ISSN: 0031-899X Journal DTLΑ English ΙT 2622-89-1, Borinic acid, diphenyl-(electronic structure, energy levels, mol. orbitals and spectrum of) RN2622-89-1 CAPLUS Borinic acid, diphenyl- (6CI, 7CI, 8CI, 9CI) (CA INDEX NAME) CN

Patel 9/24/2003>

AB Elec. dipole hyperpolarizabilities are calcd. for a large no. of 2- to 20-electron S-state atoms and ions. The calcns. are carried out within the framework of a Hartree-Fock perturbation procedure. This approxn. is developed to high order and it is shown explicitly that the total Hartree-Fock energy is obtained to 5th order from a knowledge of the Fock orbitals to 2nd order. The 1st- and 2nd-order coupled Hartree-Fock orbital equations are uncoupled in a direct manner and the resulting approx. equations solved by minimizing assocd. 1-electron functionals. The resulting hyperpolarizabilities for the He, Ne, and Ar series are all The calcd. values for the inert gases agree with expt., within a factor of 2. Some of the high-Z hyperpolarizabilities for the Na, Mg, and K series are neg., while for the Li and Be series the values are large and pos. These interesting signs are discussed. The hyperpolarizabilities are quite sensitive to the choice of zeroth order Hartree-Fock function used in the calcn.

ANSWER 236 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN L4ΑN 1966:104325 CAPLUS DN 64:104325 OREF 64:19651a-h,19652a-b TIHydroxylamine derivatives. XXVI. Acetals with N-hydroxydialkylamines ΑU Zinner, Gerwalt; Kliegel, Wolfgang CS Univ. Muenster, Germany Chemische Berichte (1966), 99(3), 895-902 CODEN: CHBEAM; ISSN: 0009-2940 DT Journal LΑ German IT 2622-89-1, Borinic acid, diphenyl-(oxime derivs.) RN 2622-89-1 CAPLUS CN Borinic acid, diphenyl- (6CI, 7CI, 8CI, 9CI) (CA INDEX NAME)

GI For diagram(s), see printed CA Issue. AB ; cf. CA 64, 19597a. N-Hydroxydialkylamines were converted with aldehydes to O-semiacetals and with amines and hydrazines to mixed O,N-acetals which were cleaved by acyl chlorides to N-acyloxydialkylamines and .alpha.-chloroamines or -hydrazines. The oximes which can be regarded as N-hydroxyimines showed a corresponding behavior including the formation of stable dihydro-5-bora-1,3,4-dioxazoles from the semiacetals and Ph2BOH (I). Et2NOH (0.050 mole) and 0.025 mole CCl3CH(OH)2 fused carefully gave 44% RCH(OH)OX (II) (X = NEt2, R = CCl3) (III), m. 69-72.degree. (petroleum ether). Similarly were prepd. II (R = CCl3, X = morpholino), 40%, m. 103-6.degree. (petroleum ether-CHCl3), and (XO)2CHR (R = CCl3, X =piperidino), 53%, m. 68.degree. (petroleum ether). III (0.050 mole) refluxed 3 hrs. in 50 cc. Et20 with a small amt. dry Na2SO4 gave 4.3 q. 1-hydroxy-morpholine (IV), b8 86.degree., n20D 1.470, and 4.1 g. 1-formylmorpholine, b7 102-6.degree., m. 22-4.degree., n25D 1.484. The

appropriate N-hydroxydialkylamine (0.1 mole) added dropwise at room temp. to 0.1 mole aq. CH2O, treated with 0.1 mole appropriate amine or hydrazine, and satd. after 1 hr. with K2CO3 yielded the corresponding R2NOCHR'NR''2 (V) listed in the 1st table. (PhCH2) NOH (0.05 mole) and 0.050 mole aq. CH2O refluxed with 50 cc. EtOH to soln., treated with 4.35 g. morpholine and 5.0 g. CaO and refluxed 6 hrs. gave 6.7 g. unreacted (PhCH2) 2NOH, m. 124.degree. and 25% (PhCH2) 2NOCH2NR'' 2 (NR''2 = morpholino), m. 71-4.degree. (petroleum ether). BzH, Et2NOH, and Et2NH (0.10 mole each) heated 30 hrs. on the water bath with 10.0 g. K2CO3 and 10.0 g. CaO and kept 1 hr. at room temp. yielded 20% V (R = R'' = Et, R' = Ph), b0.04 74.degree., n20D 1.5026. Similarly was prepd. V (R2N = R''2N = piperidino, R' = Ph) (XI), 50%, b0.001 111.degree., n20D 1.5325. Et2NOH and aq. CH2O (0.1 mole each) with 0.050 mole aq. MeNH2 gave similarly 40% (Et2NOCH2)2NMe, b9 113.degree., n20D 1.4368. In the same manner as the V were prepd. the following R2NOCHR'NR1NR22 XII (R2N, R', R1, R2, b.p./mm., n20D, and % yield given): Et2N, H, Me, Me, 70-2.degree./12,--, 40; piperidino, H, Me, Me (XIII), 105.degree./12, 1.4620, 60; piperidino, H, cyclohexyl, Me (XIV), 88-9.degree./0.01, 1.4848, 60; morpholino, H, Me, Me, 114-16.degree./15, 1.4635, 50; iso-Pr2N, H, Me, Me (XV), 88.degree./9, 1.4412, 70. IV (10.3 g.) and 5.0 g. each K2CO3 and CaO treated dropwise at room temp. successively with 7.2 g. iso-PrCOCl and 8.7 g. morpholine yielded 12.6 g. N-(2-methylpropenyl) morpholine (XVa), b11 60-58.degree., n20D 1.4461, and 8.3 g. unreacted IV, b11, 87-91.degree., n20D 1.470. A similar run with 20.6 g. IV gave 2.3 g. XVa, b13 64.degree., n20D 1.4667, 9.8 g. IV, n20D 1.470, and 16.6 g. N-(1-morpholinoisobutyloxy) morpholine, bl1 145-50.degree., b0.01 95.degree., n20D 1.4742. A few drops IV in 2 cc. EtOH heated briefly with a few drops iso-PrCHO, treated with ${\tt Ph2BOCH2CH2NH2~(XVI),~and~boiled~briefly~gave~XVII,~m.~152-4.degree.}$ (EtOH). The appropriate V or XII (0.050 mole) in 50 cc. dry Et2O treated dropwise very slowly with stirring with 0.050 suitable acyl chloride in 50 cc. dry Et20, filtered from the pptd. CICHR'NR''2 (XVIII) or ClCHR' NR1NR22 (XIX), resp., and worked up gave the corresponding R2NO2CR' (XX). In this manner were prepd. the XVIII and XX listed in the 2nd table. XIII gave similarly 80% XX (R2N = piperidino, R' = Ac), b1388.degree., and 75% ClCHMeNMeNMe2, decomp. 162.degree.. XIV yielded 79% XX (R2N = piperidino, R' = EtO2C), b12 110-12.degree., n20D 1.4530, and 100% CICH2NMeNMe2, decomp. 80-2.degree.. XV gave 85% XX (R = iso-Pr, R' = EtO2C), b11, 87-9.degree., n20D 1.4242, and 92% ClCH2NMeNMe2, m. 161.degree.. Cyclohexanone oxime (XXI) (3.5 g.) and 5.1 9. CClaCH(OH)2, refluxed 4 hrs. in 40 cc. CH2Cl2 with 5.0 g. Na2SO4 yielded 100% O-(2,2,2-trichloro-1hydroxy-ethyl)cyclohexanone oxime, m. 107.degree. (CH2Cl2-petroleum ether). XXI (11.3 g.) in 0.1 mole aq. CH2O treated with cooling and stirring with 0.050 mole aq. MeNH2 and satd. after 1 hr. with K2CO3 yielded 46% N, N-bis(cyclohexylideneaminooxymethyl) methylamine, b0.05 127-30.degree., n20D 1.5027. XXI, piperidine, and aq. CH2O(0.1 mole each) yielded similarly 76% O-piperidinomethyl deriv. (XXII) of XXI, b0.01 80.degree., bl0 143-5.degree., n20D 1.4957. XXII cleaved with BzCl in dry Et20 gave 100% OBz deriv. (XXIII) of XXII, m. 63.degree., and 100% 1-chloromethylpiperidine. XXI, aq. CH2O, and aq. Me2NH (0.1 mole each) gave similarly 65% OMe2NCH2 deriv. of XXI, bl2 97.degree., n25D 1.4737. XXI, aq. CH2O, and MeNH-NH2 (0.1 mole each) yielded 57% OMe2NNMe deriv. (XXIV) of XI, b0.01 74.degree., b36 145-50.degree. n20D 1.4790, which cleaved with BzCl gave 90% XXIII, m. 64.degree., and 100% ClCH2NMeNMe2, m. 162.degree.. A small amt. XXI, 2 cc. EtOH, and 0.5 cc. aq. CH2O heated briefly, treated with a small amt. XVI, and boiled briefly again yielded XXV (X = cyclohexylidene), m. 137-8.degree.. Me2C:NOH gave similarly XXV (X = Me2C:), m. 148-9.degree. (EtOH).

Patel

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10085368.2
                               Page 315
L4
     ANSWER 237 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN
AN
     1966:71259 CAPLUS
DN
     64:71259
OREF 64:13369c-d
ΤТ
     Analytical spectroscopic investigation of the relation between structure
     and complexing capacity of certain metal-chelating agents in the
     azomethine series
     Umland, F.; Poddar, B. K.; Stegemeyer, H.
ΑU
CS
     Univ. Muenster, Germany
SO
     Zeitschrift fuer Analytische Chemie (1966), 216(1), 125-50
     CODEN: ZANCA8; ISSN: 0372-7920
DT
     Journal
LΑ
     German
IT
     2622-89-1, Borinic acid, diphenyl-
        (electronic structure, hydrogen bonds, ionization and spectra of)
     2622-89-1 CAPLUS
RN
     Borinic acid, diphenyl- (6CI, 7CI, 8CI, 9CI) (CA INDEX NAME)
CN
   Ph
Ph-B-OH
AB
     A no. of o-mono-and o,o'-disubstituted azo-methines were prepd. and their
     complexes with Cu, Zn, and Cd were studied. From the ir spectra a
     relation between intramol. H bond, ring size, and stability of the
     chelates was derived. Six-membered chelate rings are far more stable than
     5-membered rings. Five-membered chelate rings are formed only when
     stabilized by a 6-membered ring in the same chelate.
                                                          From the C:N
     absorption band it follows that in 6-membered rings mainly the free
     electron pair of the N participate in chelation. In 5-membered chelates,
     however, the .pi.-electrons of the double bond also participate.
     Tridentate chelate reagents form 1:1 chelates with 1 coordination position
     unoccupied which is subject to synergetic effects by monodentate ligands
     in liquid-liquid extn. Useful sepns. by extn. and detns. by photometry
     can be performed by using these synergetic effects.
L4
     ANSWER 238 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN
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1966:63296 CAPLUS

AN

DN 64:63296

OREF 64:11863h,11864a-c

Photometric determination of diphenylborinic acid and its esters with diphenylcarbazone

ΑU Thierig, D.; Umland, F.

CS Westfaelischen Wilhelms-Univ., Muenster, Germany

SO Zeitschrift fuer Analytische Chemie (1966), 215(1), 24-30 CODEN: ZANCA8; ISSN: 0372-7920

DTJournal

LΑ German

ΙT 2622-89-1, Borinic acid, diphenyl-(detn. of, diphenylcarbazone in)

RN 2622-89-1 CAPLUS

CN Borinic acid, diphenyl- (6CI, 7CI, 8CI, 9CI) (CA INDEX NAME) Ph | | Ph— B— OH

(esters, detn. of, diphenylcarbazone in. ΑB The blue 1:1 diphenylcarbazone-Ph2BOH complex is catalyzed by the addn. of 5% PhOH and has an absorption max. at 590 m.mu. with a molar absorptivity of 4 .times. 104 1./mole-cm. in C6H6 soln. The complex is analyzed at its max. in the range 1-40 .gamma. in 10 ml. of C6H6 soln. with a standard deviation of 0.12 .gamma. and a variance of 0.9%. The PhB(OH)2 complex has an absorption max. at 560 m.mu. with 1/60 the intensity of the diphenyl complex. The solvent has a strong effect on the intensity of the colors. For example, 10-4-10-5M solns. gave only weak blue or no color when 50% of MeOH, tetrahydrofuran, or dioxane was added to the C6H6 solns. of the complexes. 8-Hydroxyquinolinium tetraphenylborate is dissolved in hot AcOH, boiled 1 min., and cooled to ice temp. to give, upon drying at 100.degree. 8-hydroxyquinolinato-(O,N-B)-diphenylboron, m. 203-4.degree. (EtOH). Mixing 150 ml. of an alc. soln. contg. 950 mg. of diphenylborinic. acid, aminoethyl ester with 300 ml. of a soln. contg. 430 mg. of bis(2-pyridyl)glycol gives on standing 12 hrs. at room temp. a white ppt., which, upon washing with 50 ml. of EtOH and drying at 80.degree. is pure bis(2-pyridyl)-glycolatobis[(O,N-B)-diphenylboron], m. 267-8.degree.turning orange above 200.degree.. A soln. of 700 mg. of Pb(OAc)2.3H2O and 470 mg. of dimethylglyoxime in 30 ml. of hot HOAc is treated with 2 g. of NaBPh4 boiled 3 min., and cooled. A 25% yield of bis[(O,O-B)diphenylboron], m. 307.degree. (dichloroethane).

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L4
     ANSWER 239 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN
AN
     1966:59376 CAPLUS
DN
     64:59376
OREF 64:11063g-h,11064a-b
ΤI
     A nuclear magnetic resonance study of hydrogen bonding in
     {\sf tris}(2-N-{\sf methylaminoethyl}) borate and {\sf similar} compounds
ΑU
     Meek, Devon W.; Springer, Charles S., Jr.
CS
     Ohio State Univ., Columbus
SO
     Inorg. Chem. (1966), 5(3), 445-50
DT
     Journal
LΑ
     English
IT
     2622-89-1, Borinic acid, diphenyl-
        (electronic structure, spectrum of)
RN
     2622-89-1 CAPLUS
```

Borinic acid, diphenyl- (6CI, 7CI, 8CI, 9CI) (CA INDEX NAME)

Ph | Ph— B— OH

CN

AB B(OCH2CH2NH2)3 and the analogous N-methylamino and N,N-dimethylamino compds. were prepd. by the transesterification of Me borate with the appropriate 2-aminoethanol. The N.M.R. spectra show that extensive assocn. of the terminal amino groups occurs in pure tris(N-methylaminoethyl) borate and that this assocn. can be broken apart by heating to 160.degree. or by dissoln. in polar org. solvents such as triethylamine or MeCN. In order to det. whether the assocn. results from H bonding or internal B-N coordination, several model systems were

investigated. The spectra of NH2C2H5.BF3, piperidine.BF3, and Ph2BOCH2CH2NH2 in MeCN contain very complicated NCH2 peaks and broad NH peaks which appear at low applied magnetic field (.tau. 5.26-5.56). The broadening of the NH peak in the B-N adducts is attributed to the effect of the 14N quadrupole, whereas the complex splitting of the NCH2 multiplet is attributed to coupling with 11B in the dative bond with N and possibly with the N protons. The spectra of B(OCH2CH2NH2)3 and B(OCH2CH2NHCH2)2, on the other hand, show sharp NH peaks at .tau. 7.84, 6.78 and contain 2 sharp triplets attributed to the two sets of methylene protons in the -OCH2CH2N< units (.tau.OCH2 6.26-6.55; .tau.NCH2 7.23-7.57). The N-H peak of B(OCH2CH2NHCH3)3 appears as a sharp singlet in MeCN solns. owing to rapid exchange of the amine proton. Evidence is presented that the exchange is catalyzed by a trace (<8.7 ppm.) of water in the hygroscopic solvent, MeCN, even after rigorous drying. All of the data indicate that intermol. H bonding strongly predominates over N .fwdarw. B dative bonding in the association of B(OCH2CH2NHCH3)3.

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L4
    ANSWER 240 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN
AN
     1966:27646 CAPLUS
DN
     64:27646
OREF 64:5125q-h,5126a-b
ΤI
     Reaction of .beta.-oxo enols with diphenylborinic esters
ΑU
     Bally, Ioana; Arsene, A.; Bacescu-Roman, Maria; Balaban, A. T.
CS
     Inst. At. Phys., Bucharest, Rom.
SO
     Tetrahedron Letters (1965), (44), 3929-31
     CODEN: TELEAY; ISSN: 0040-4039
DT
     Journal
LΑ
     English
IT
     2622-89-1, Borinic acid, diphenyl-
        (esters, reaction with .beta.-oxo enols)
RN
     2622-89-1 CAPLUS
     Borinic acid, diphenyl- (6CI, 7CI, 8CI, 9CI)
CN
                                                   (CA INDEX NAME)
```

Ph | Ph-B-OH

GI

cf. CA 64, 4902e; preceding abstr. Ph2BOCH2CH2NH2 and .beta.-oxoenols (1,3-diketones or .omicron.-acylphenols) refluxed 5-10 hrs. in C6H6, the cooled soln. (dild. with petroleum ether) filtered and the stable compds. recrystd. from C6H6, MeCN, or alc. gave the yellow cryst. products (I or II) as tabulated (formula, R1, R2, R3, m.p., .lambda. max. in m.mu. (MeCN) given): I, Ph, H, Ph, 227.degree. 391, 309, 300, 279sh; I, Ph, H, p-MeOC6H4, 231.degree. (decompn.), 400, 334, 309, 300, 284; I, p-MeOC6H4, H, p-MeOC6H4, 283.degree., 420, 404, 314, 269; I, Me, H, p-MeOC6H4, 209.degree., 365, 320, 310, 270; I, Me, Me, Me, 135.degree., 338, 310, 278sh; II, H, --, --, 149.degree., 375, 309, 280; Me, --, --, 115.degree. (decompn.), 364, 309, 275. In KBr pellets, the ir spectra gave one band at 1595-1620 cm.-1 in the range 1600-2000 cm.-1 due to aryl groups. Bands due to B-O stretching vibrations appeared at 1300-1370 cm.-1 The products I have the same skeleton as previously described compds. with R1 = R2 = F,

L4 ANSWER 241 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN AN 1965:439178 CAPLUS

R1 = R2 = alkyl, and R1R2 = 1,2-benzodioxa (CA 61, 10610e).

For diagram(s), see printed CA Issue.

63:39178 OREF 63:7028g-h,7029a Polyfluoroaryl organometallic compounds. II. Pentafluorophenylboron ΤI halides and some derived compounds Chambers, R. D.; Chivers, T. ΑU Univ. Durham, UK CS SO Journal of the Chemical Society, Abstracts (1965) 3933-9 CODEN: JCSAAZ; ISSN: 0590-9791 DT Journal LΑ English

IT 2118-02-7, Borinic acid, bis(pentafluorophenyl)(prepn. of)

RN 2118-02-7 CAPLUS

CN Borinic acid, bis(pentafluorophenyl) - (7CI, 8CI, 9CI) (CA INDEX NAME)

cf. CA 62, 6501d. Pentafluorophenylboron dihalides C6F5BX2 (X = Cl or F) are prepd. by cleavage of trimethylpentafluorophenyltin, dimethylbis(penta-fluorophenyl)tin and methylpentafluorophenylmercury with boron halides; and bis(pentafluorophenyl)boron chloride, (C6F5)2BCl, can be obtained by reaction of dimethylbis(pentafluorophenyl)tin with 1 mol. BCl3. Unlike the perfluoroalkyl or perfluorovinyl compds., the perfluoroaryl derivs. do not decomp. by migration of F from C to B; on heating, the dihalides disproportionate; 2C6F5BX2 .fwdarw. (C6F5)2BX + BX3 (X = Cl or F). Careful hydrolysis of the di- and monohalides gave pentafluorophenylboronic acid and bis(pentafluorophenyl)borinic acid, resp. These acids show an unusual susceptibility to nucleophilic cleavage of the org. groups and a resistance to dehydration. The N.M.R. spectra of pentafluorophenylboron compds.indicate .pi.-p interaction in the tricovalent boron derivs.

L4ANSWER 242 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN ΑN 1965:422178 CAPLUS DN 63:22178 OREF 63:3894a-c ΤI The electrochemical oxidation of white phosphorus ΑU Barry, Michael L. CS Univ. of California, Berkeley SO (1964), AEC Accession No. 5589, Rept. No. UCRL-11573, 155 pp. Avail.: OTS From: Nucl. Sci. Abstr. 19(3), 627 (1965).

DT Report

LA English

CN Borinic acid, diphenyl- (6CI, 7CI, 8CI, 9CI) (CA INDEX NAME)

The electrochem. oxidn. of liquid and solid white P impregnated in porous AΒ conducting matrixes in alk., neutral, and acidic electrolytes was investigated. Rates of the spontaneous disproportionation-decompn. reactions of P and the simple P oxyacids in these electrolytic solns. were also measured. Coulometric studies of the P electrode were made by comparing the rates of formation of H3PO4, H3PO3, H3PO2, PH3, and H at known c.ds. to the rates of formation at zero c.d. The half-cell potentials measured for the P electrodes (relative to the standard H electrode) ranged from 1.08 v. in 10N NaOH to 0.00 v. in 10N H2SO4 compared to theoretical values of 1.83 v. and 0.48 v. for the oxidn. of P to phosphite or H3PO3 in these solns. In alk. media the electrode potentials were actually established by PH3 produced from the disproportionation-decompn. reactions of P. The reasons for the low electrode potentials in acidic media are not known. In a Ag amalgam matrix in neutral and acidic media, P was quant. oxidized to H3PO3 and H3PO4 with overall coulombic efficiencies approaching 100% at c.ds. up to 10 ma./cm.2 The coulometric results in alk. media were obscured by the effects of the simultaneous disproportionation-decompn. reactions of P.

L4 ANSWER 243 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

AN 1964:494425 CAPLUS

DN 61:94425

OREF 61:16476q-h

TI Triarylborane complexes, a new series of broad-spectrum germicides

AU Updegraff, D. M.

CS Minnesota Mining & Manufg. Co., St. Paul, MN

SO Journal of Infectious Diseases (1964), 114, 304-10

CODEN: JIDIAQ; ISSN: 0022-1899

DT Journal

LA Unavailable

IT 12113-07-4, Sodium hydroxytriphenylborate

(bactericidal, fungicidal and protozoacidal action of)

RN 12113-07-4 CAPLUS

CN Borate(1-), hydroxytriphenyl-, sodium, (T-4)- (9CI) (CA INDEX NAME)

● Na+

AB More than 100 coordination complexes of triarylboranes with amines and substituted phosphines were screened against bacteria and fungi, and

selected members were also screened against protozoa. The chem. stable complexes of triphenylborane and tris(para-substituted phenyl)borane were powerful broad-spectrum germicides, fungicides, and protozoicides.

L4 ANSWER 244 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

AN 1964:494424 CAPLUS

DN 61:94424

OREF 61:16476f-g

TI Effect of L-cysteine on .alpha.-amylase production and nucleic acid metabolism in Bacillus subtilis

AU Oishi, Michio; Kitayama, Shigeru; Takahashi, Hajime; Maruo, Bunji

CS Univ. Tokyo

SO Journal of General and Applied Microbiology (1963), 9(3), 337-41 CODEN: JGAMA9; ISSN: 0022-1260

DT Journal

LA Unavailable

RN 12113-07-4 CAPLUS

CN Borate(1-), hydroxytriphenyl-, sodium, (T-4)- (9CI) (CA INDEX NAME)

• Na+

RN 95493-42-8 CAPLUS

CN Borinic acid, diphenyl-, sodium salt (6CI, 7CI) (CA INDEX NAME)

Na

AB In B. subtilis, 10-4M L-cysteine caused an almost complete inhibition of adenine incorporation into ribo- and deoxyribonucleic acids and 2 .times. 10-5M amts. suppressed .alpha.-amylase production to 80-90% of the controls. The latter inhibition was reversed by unidentified compds. in yeast ext.

L4 ANSWER 245 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

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10085368.2
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Page 321

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AN
     1964:90951 CAPLUS
     60:90951
DN
OREF 60:15899b-e
     A novel disproportionation of arylboronic acids
TI
ΑU
     Dewar, Michael J. S.; Dougherty, Ralph C.
     Univ. of Texas, Austin
CS
SO
     Tetrahedron Letters (1964), (15-16), 907-11
     CODEN: TELEAY; ISSN: 0040-4039
DT
     Journal
LΑ
     Unavailable
IT
     2622-89-1, Borinic acid, diphenyl-
        (prepn. and storage of)
     2622-89-1 CAPLUS
RN
CN
     Borinic acid, diphenyl- (6CI, 7CI, 8CI, 9CI)
                                                    (CA INDEX NAME)
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GI

AB In an attempt to synthesize 4,6-dimethyl-2-phenyl-2,3-borazaropyridine (I), 20 g. triphenylboroxine and 20 g. acetylacetone was added to a mixt. of 2.5 g. tert-BuOK and PhMe in a Dean-Stark app., and refluxed 24 hrs. in a current of NH3 till no H2O was evolved to give 12.4 g. 2,2-diphenyl-4,6-dimethyl-1H-boroxazine (II), m. 101-1.5.degree., whose structure was evidenced by its infrared and nuclear magnetic resonance spectra. II arose by disproportionation of phenylboronic acid to diphenylborinic acid. This mechanism was confirmed by a synthesis of II from diphenylborinic acid, acetylacetone, and NH3 in 82% yield, and by an expt. in which triphenylboroxine was heated alone with tert-BuOK in xylene; diphenylborinic acid was isolated as its ethanolamine deriv. Similarly, tri-o-tolylboroxine and tert-BuOK in xylene gave di-o-tolylborinic acid, characterized by its reaction with acetylacetone and NH3 to give 71% 2,2-di(o-tolyl)-4,6-dimethyl-1H-boraxazine, m. 125-5.5.degree.. Its structure was consistent with its nuclear magnetic resonance spectrum. This disproportionation was a general reaction for

arylboronic acids; alkylboronic acids were recovered. The mechanism of

L4 ANSWER 246 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN AN 1964:90950 CAPLUS

the reaction was discussed.

For diagram(s), see printed CA Issue.

DN 60:90950

```
OREF 60:15899a-b
     Preparation and storage of diphenylborinic acid and its anhydride
ΑU
     Chremos, G. N.; Weidmann, H.; Zimmerman, H. K.
CS
    A. & M. Coll. of Texas, College Station
SO
     Journal of Organic Chemistry (1961), 26(5), 1683
     CODEN: JOCEAH; ISSN: 0022-3263
DT
     Journal
LΑ
    Unavailable
TT
     2622-89-1, Borinic acid, diphenyl-
        (prepn. and storage of)
RN
     2622-89-1 CAPLUS
     Borinic acid, diphenyl- (6CI, 7CI, 8CI, 9CI) (CA INDEX NAME)
CN
```

AB The ethanolamine ester (I) of diphenylborinic acid (II) may be prepd. and kept up to 3 years without decompn. When free II was required, I in MeOH was hydrolyzed by addn. of M HCl. II was extd. with petr. ether (30-60.degree.), washed with M HCl and distd. H2O, dried, and evapd. until the diphenylborinic anhydride pptd.

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L4
    ANSWER 247 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN
AN
    1964:64120 CAPLUS
DN
     60:64120
OREF 60:11306h,11307a
    Arylboric acids X. Action of some organoboron compounds on wheat roots and
TI
     on pollen and distribution of 14C-labeled benzeneboronic acid in plants
ΑU
     Torssell, Kurt
    Univ. Stockholm
CS
SO
     Physiologia Plantarum (1963), 16(1), 92-103
     CODEN: PHPLAI; ISSN: 0031-9317
DT
    Journal
LΑ
    Unavailable
IT
     2622-89-1, Borinic acid, diphenyl-
        (plant regulator action of)
RN
     2622-89-1 CAPLUS
```

Borinic acid, diphenyl- (6CI, 7CI, 8CI, 9CI) (CA INDEX NAME)

CN

AB cf. CA 54, 22437h. Isobutane- and hexaneboronic acids slightly stimulated root elongation at 10-5M, but diphenyl- and dianisylborinic acids did not and were toxic at higher concns. The bifunctional m- and p-benzenediboronic acids had peaks of stimulation at 10-5M, but then became toxic. Flax, pea, and cress responded in the same general way. Benzeneboronic acid (I) had slight stimulation for growth of pollen of Tulipa fosteriana. 14C-I or its degradation products were quickly and uniformly distributed throughout the bean plant. After 18 hrs. only traces of unchanged I could be detected.

ANSWER 248 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN L4 AN 1964:27474 CAPLUS DN 60:27474 OREF 60:4851g The acidity of diphenyl hydroxyborane ΤI Chremos, George N.; Zimmerman, Howard ΑU CS Case Inst. Technol., Cleveland, OH Chim. Chronika (Athens, Greece) (1963), 28(9), 103-7 SO DT Journal LΑ English ΙT 2622-89-1, Borinic acid, diphenyl-(reactions and spectrum of) RN 2622-89-1 CAPLUS Borinic acid, diphenyl- (6CI, 7CI, 8CI, 9CI) (CA INDEX NAME) CN

AB The degradation of diphenyl hydroxyborane (I) in H2O and aq. dioxane at 25.degree. was studied. The reaction was followed by the disappearance of a ultraviolet absorption max. at 234 m.mu. and appearance of a weak absorption at 266 m.mu.. The rates were calcd. and showed that the reaction was 1st order. A mechanism was suggested which successfully predicted the use of HCl for the stabilization of aq. solns. of I. Protolysis dissocn. consts. at 15-55.degree. for I were also detd.

L4 ANSWER 249 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

AN 1963:462546 CAPLUS

DN 59:62546

OREF 59:11557c-e

TI Aryl polyboronic acids and esters

IN Washburn, Robert M.; Billig, Franklin A.

PA American Potash & Chemical Corp.

SO 5 pp.

PΙ

DT Patent

LA Unavailable

PATENT NO. KIND DATE APPLICATION NO. DATE

US 3090801 19630521 US 19560706

IT 66117-64-4, Borinic acid, di-p-tolyl-

(prepn. of) RN 66117-64-4 CAPLUS

CN Borinic acid, bis(4-methylphenyl) - (9CI) (CA INDEX NAME)

AB Aryl Na compds. (I) react smoothly with borate esters (II) to give high yields of areneboronic acids, diareneborinic acids, or triarylborines. I,

which were not stable, were preferably prepd. from the aromatic halide (III) and finely-dispersed metallic Na (IV). I can be added to II, II can be added to I, or III, II, and IV can be combined together. In an example, 23 g. Na in 23 g. xylene added to 56.3 g. PhCl and 155.9 g. (MeO) 3B at a rate to keep the temp. at 25-30.degree., treated with MeOH and H2O to hydrolyze PhB(OMe) 2, azeotropically distd. with H2O to remove xylene, MeOH, and reaction products gave, upon cooling the residue, 41% PhB(OH) 2, m. 216.degree.

L4ANSWER 250 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN ΑN 1963:33497 CAPLUS DN 58:33497 OREF 58:5714e-g Reactions of ephedrine bases with diphenylboric acid. I. Constellation of TI alkanolamines ΑU Roth, H. J.; El Din, N. Nour Tech. Hochschule, Braunschweig, Germany CS SO Arch. Pharm. (1962), 295, 679-89 DT Journal Unavailable LΑ IΤ 2622-89-1, Borinic acid, diphenyl-(prepn. of) 2622-89-1 CAPLUS RN Borinic acid, diphenyl- (6CI, 7CI, 8CI, 9CI) (CA INDEX NAME) CN

Ph | Ph— B— OH

GI For diagram(s), see printed CA Issue.

The ephedrine (I) base (0.005 mole) in 10 ml. MeOH was treated with 10 ml. 0.007 molar Ph2BOH in MeOH and the soln. concd. on a water bath to 1/2 vol. to give the corresponding 2,2-diphenylboroxazolidine [I base, % yield, m.p., and Rf value (6:1:1 BuOH-EtOH-H2O) given]: norephedrine, 80, 185.degree., 0.67; pseudonorephedrine, 82, 258-61.degree., 0.70; I, 78, 221-2.degree., 0.73; pseudoephedrine, 80, 178-9.degree., 0.68; N-methylephedrine, 90, 207-8.degree., 0.65; N-methylpseudoephedrine, 96, 184.degree. 0.72. The rate of formation of the boraxazotidine derivs. of the ephedrine compds. was markedly lower than that of the pseudo series. It was suggested that in alc. soln. in the presence of Ph2BOH and alk. the pseudo bases had the constellation II while the normal series had the constellation III.

L4 ANSWER 251 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN AN1963:33496 CAPLUS DN 58:33496 OREF 58:5714d-e Catalytic dehydrocondensation of phenyldichlorosilane with benzene ΤI ΑU Mal'nova, G. N.; Mikheev, E. P. SO Plasticheskie Massy (1962), (8), 20-3 CODEN: PLMSAI; ISSN: 0554-2901 DTJournal LΑ Unavailable IT 2622-89-1, Borinic acid, diphenyl-(prepn. of) RN 2622-89-1 CAPLUS

CN Borinic acid, diphenyl- (6CI, 7CI, 8CI, 9CI) (CA INDEX NAME)

Dehydrocondensation occurs between PhSiHCl2 (I) and benzene in the presence of boric acid catalyst. When a 1:1 ratio of I to benzene is heated in an autoclave for 5.5 hrs. at 250.degree., the pressure reaches 103 atm. and remains steady. The H yield is 0.31 mole/mole of I, and the yield of Ph2SiCl2 (II) is 20%. The known disproportionation of I permits dehydrocondensation of benzene with the H-contg. disproportionation products. An approx. calcn. indicates that about 70% of the yield of II is derived from condensation of I with benzene, and about 30% results from the disproportionation of I. The side reactions are diminished by diln. with benzene. E.g., a 1:3 ratio of I and benzene, when heated to 260.degree. for 4.5 hrs., produces 0.46 mole H/mole I, and the yield of II is increased to 30%.

L4 ANSWER 252 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

AN 1963:27392 CAPLUS

DN 58:27392

OREF 58:4589e-g

TI Possible quantitative coupling of radicals bound to a central atom of an element. II. Organoboron compounds

AU Lapkin, I. I.; Yuzhakova, G. A.

CS State Univ., Perm

SO Zhurnal Obshchei Khimii (1962), 32, 1967-9 CODEN: ZOKHA4; ISSN: 0044-460X

DT Journal

LA Unavailable

RN 20631-84-9 CAPLUS

CN Borinic acid, bis(2,4,6-trimethylphenyl) - (9CI) (CA INDEX NAME)

GI For diagram(s), see printed CA Issue.

cf. CA 55, 14346b. BF3-Et2O and 3 moles 2,4,6-Me3C6H2MgBr in Et2O gave dimesitylboronic acid, m. 141.degree.; when the reaction was run in refluxing MePh 8 hrs., the product was (2,4,6-Me3C6H2)3B, m. 193.degree.. Similarly was prepd. 21-76% I (R and m.p. given): Me, 262.degree.; Et, 217.degree.; PrO, 176.degree.; Bu, 140.degree.; Am, 114.degree.; iso-Pr, 218.degree.; iso-Bu, 154.degree.; and iso-Am, 102.degree. No bis(2-alkoxy-1-naphthyl)boronic acids could be isolated from these reactions, owing to facile symmetrization of such substances. Conformational analysis of the trimesitylboron showed that the 3 aromatic rings must exist out of planar configuration. The above alkoxynaphthyl

derivs. were not oxidized by air at room temp.; the same was true also of di(o-substd. triaryl) boron derivs. The latter do not react with NH3, amines or ethers.

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ANSWER 253 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN
L4
AN
     1963:14982 CAPLUS
DN
     58:14982
OREF 58:2471q-h
ΤI
    Arylboronic and diarylborinic acids and their esters and anhydrides
     Rosinger, Herbert P.; Yates, John; Wright, William E.
IN
PΑ
     "Shell" Research Ltd.
SO
     6 pp.
DT
     Patent
LΑ
     Unavailable
                     KIND DATE
     PATENT NO.
                                           APPLICATION NO. DATE
    GB 906145
PΙ
                            19620919
                                           GB
                                                            19591130
     2622-89-1, Borinic acid, diphenyl-
IT
        (prepn. of)
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Borinic acid, diphenyl- (6CI, 7CI, 8CI, 9CI) (CA INDEX NAME)

Ph | | Ph— B— OH

2622-89-1 CAPLUS

RN

CN

AP Arylboron compds. are prepd. by treating an arylmagnesium chloride with a boric acid triester. In an example Mg turnings 13.6 parts were treated under anhyd. conditions under N with a soln. of PhCl 56.4 in dry redistd. xylene 170 parts by vol. together with a crystal of iodine. The mixt. was stirred and refluxed 5 hrs. Xylene 180 was added and the resulting Grignard suspension added slowly to a soln. of 60 parts Me borate in 160 parts by vol. xylene at -30.degree. under N and anhyd. conditions. The mixt. was stirred 2 hrs., then hydrolyzed at 10.degree. with 20% H2SO4 300 parts. Extn. of the aq. layer with xylene gave phenylboronic acid anhydride. In other examples Pr borate, Bu borate, and iso-Bu borate were used. The 2-aminoethyl ester of diphenylborinic acid was also prepd.

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L4
    ANSWER 254 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN
     1962:73541 CAPLUS
ΑN
DN
     56:73541
OREF 56:14311a-c
     Deboronation: Formation of phenylboronic anhydride from
     diphenylhydroxyborane in the presence of amides
AU
     Zimmerman, Howard K., Jr.
CS
    A. & M. Coll. of Texas, College Station
SO
     Journal of Organic Chemistry (1961), 26, 5214-15
     CODEN: JOCEAH; ISSN: 0022-3263
DT
     Journal
LΑ
    Unavailable
IT
     2622-89-1, Borinic acid, diphenyl-
        (reaction with amides)
RN
     2622-89-1 CAPLUS
     Borinic acid, diphenyl- (6CI, 7CI, 8CI, 9CI) (CA INDEX NAME)
CN
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Diphenylhydroxyborane (I) treated in PhMe gave 44% recovery of bis(diphenylboron) oxide (II). No phenylboronic anhydride was formed. AcNH2 (0.8 g.) with 450 ml. PhMe treated with I from 3 g. di-B-phenylboroxazolidine (III), the mixt. azeotropically distd., and the residue crystd. overnight gave 1.3 g. phenylboronic anhydride (IV), m. 214-16.degree. IV also characterized by prepn. from N-ethyl-B-phenyl-diptychboroxazolidine. PhMe (200 ml.) I (from 3 g. III) azeotropically distd. and crystd. gave 1.85 g. IV and benzamide. Phthalimide (1.3 g.) with 200 ml. PhMe azeotropically distd. with I (from 2 g. III) gave 1.2 g. phthalimide, and the filtrate gave 0.1 g. IV. I(3g.) in 160 ml. PhMe distd. to 3-5 ml. after 4 hrs. gave 1 g. II, m. 116-18.degree.. The results suggested that the crucial property required of a deboronating agent may be its Lewis base character rather than any oxidizing power per se.

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ANSWER 255 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN
L4
AN
     1962:66979 CAPLUS
     56:66979
DN
OREF 56:12918d-e
ΤI
     Aminoditolyborane and the preparation of diarylborinic acids
ΑU
     Coates, G. E.; Livingstone, J. G.
CS
     Durham Coll., UK
SO
     Journal of the Chemical Society, Abstracts (1961) 4909-11
     CODEN: JCSAAZ; ISSN: 0590-9791
DT
     Journal
LΑ
     Unavailable
IT
     2622-89-1, Borinic acid, diphenyl- 62981-91-3, Borinic
     acid, di-1-naphthyl- 66117-64-4, Borinic acid, di-p-tolyl-
     73774-44-4, Borinic acid, di-o-tolyl- 89566-59-6,
     Borinic acid, bis(p-chlorophenyl) - 96484-29-6, Borinic acid,
     bis (p-bromophenyl) -
        (prepn. of)
RN
     2622-89-1 CAPLUS
CN
     Borinic acid, diphenyl- (6CI, 7CI, 8CI, 9CI) (CA INDEX NAME)
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Ph
|
Ph— B— OH
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RN 62981-91-3 CAPLUS CN Borinic acid, di-1-naphthalenyl- (9CI) (CA INDEX NAME)

RN 66117-64-4 CAPLUS

CN Borinic acid, bis(4-methylphenyl) - (9CI) (CA INDEX NAME)

RN 73774-44-4 CAPLUS

CN Borinic acid, bis(2-methylphenyl) - (9CI) (CA INDEX NAME)

RN 89566-59-6 CAPLUS

CN Borinic acid, bis(4-chlorophenyl) - (9CI) (CA INDEX NAME)

RN 96484-29-6 CAPLUS

CN Borinic acid, bis(4-bromophenyl) - (9CI) (CA INDEX NAME)

AB The following route to diarylborinic acids was investigated: BCl3 + Ph2NH .fwdarw. Cl2BNPh2 (I). I + ArMgX .fwdarw. Ar2BNPh2 (II). II + H2O + H2NCH2CH2OH .fwdarw. Ar2BOCH2CH2NH2 (III). III + HCl .fwdarw. Ar2BOH. Only a slight excess of Grignard reagent is used, making this procedure more economical than present ones. The yields of III are in the range of 51-93% and there is no contamination by boronic acids. The following new acids and their 2-aminoethyl esters were prepd. (R in R2BOR'); o-MeOC6H4, m.p. of ester 164-5.degree.; PhC.tplbond.C, m.p. of ester 172-4.degree., of acid 98-100.degree.; 3,4-Me2C6H3, m.p. of ester 204-6.degree.

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L4
    ANSWER 256 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN
AN
     1962:53528 CAPLUS
DN
     56:53528
OREF 56:10187c-i,10188a
ТT
     Organic compounds of boron and phosphorus
IN
     Birum, Gail H.; Dever, James L.
PA
    Monsanto Chemical Co.
DT
     Patent
LΑ
    Unavailable
     PATENT NO.
                     KIND DATE
                                          APPLICATION NO.
                                                           DATE
                           -----
                                          -----
    US 3014952
PΙ
                           19611220
                                                           19590320
     2622-89-1, Borinic acid, diphenyl-
ΙT
        (esters, with di-Et (hydroxyalkyl)phosphonates)
     2622-89-1 CAPLUS
RN
CN
     Borinic acid, diphenyl- (6CI, 7CI, 8CI, 9CI) (CA INDEX NAME)
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AΒ Phosphinyl esters of boron acids, useful as insecticides, lubricant additives, plasticizers, flameproofing agents, and especially for inhibiting the preignition of leaded fuels, were prepd. by the reaction of a B compd. contg. a halogen atom, a carbonyl compd., and an alkyl, or haloalkyl phosphite, phosphonite, or phosphinite. In an example, 32.0 g. 4,4,6-trimethyl-2-chloro-1,3,2-dioxaborinane added to 32.8 g. P(OEt)3 and 11.4 g. EtCHO dropwise during 10 min. at 15-20 degree., the mixt. allowed to reach room temp., heated to 50.degree., the EtCl removed in vacuo, and the residue concd. to 100.degree./0.1 mm. gave 97.7% 2-[1-(diethoxyphosphinyl)propoxy]-4,4,6-trimethyl-1,3,2-dioxaborinane, n25D 1.4346. Likewise prepd. were the following (product, % yield, n25D given): 2-[1-(diethoxyphosphinyl)propoxy]-5,5-dimethyl-1,3,2dioxaborinane, 99.3, 1.4415, b0.1-0.15 128-30.degree.; 2-[1-(diethoxyphosphinyl)ethoxy]-5,5-dimethyl-1,3,2-dioxaborinane, -, 1.4400, b0.05 lll-14.degree.; 2-[.alpha.-(diethoxyphosphinyl)furfuryloxy]-5-ethyl-5-methyl-1,3,2-dioxaborinane, 99, 1.4727; 2[1-(diethoxyphosphinyl)cyclohexyloxy] -5-ethyl-5-methyl-1,3,2-dioxaborinane, 99, 1.4651; 2-[1-(dimethoxyphosphinyl)propoxy]-4-methyl-1,3,2dioxaborinane, 99.3, 1.4420; 2-[.alpha.-(diethoxyphosphinyl)-2chlorobenzyloxy] - 5-ethyl-5-methyl-1,3,2-dioxaborinane, -, 1.4952; 2-(1-diethoxyphosphinyl)propoxy]-5-ethyl-4-propyl- 1,3,2-dioxaborinane, -, 1.4413; 2-[1-(dimethoxyphosphinyl)-3-carbethoxypropoxy]-1,3,2dioxaborinane, 93, 1.4521; 2-[.alpha.-(dimethoxyphosphinyl)-.alpha.methylbenzyloxy]-4-methyl-1,3,2-dioxaborinane, 94, 1.5048; 2-[2-(diethoxyphosphinyl)-2-propoxy]-1,3,2-dioxaborolane, -, -;

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2-[1-(diethoxyphosphinyl)propoxy]-4-methyl-1,3,2-dioxaborinane, 100,
     1.4372; 2-[1-(diethoxyphosphinyl)-2,2,2-trichloroethoxy]-5-
     ethyl-5-methyl-1,3,2-dioxaborinane, 97, 1.4770; 2-[.alpha.-
     (diethoxyphosphinyl)-4-methylbenzyloxy]-5-ethyl-5- methyl-1,3,2-
     dioxaborinane, -, 1.4913; 2-[2-(diethoxyphosphinyl)propoxy]-1,3,2-
     dioxaborinane, -, 1.4438; tris[1-(diethoxyphosphinyl)ethyl] borate, -,
     1.4368; tris[1-(diethoxyphosphinyl)propyl] borate, -, -;
     tris[1-(diisopropoxyphosphinyl)propyl] borate, -, 1.4347;
     tris[1-[bis(2-chloroethoxy)phosphinyl]ethyl]borate, -, -; 2-chloropropyl
    bis[1-(dihexyloxyphosphinyl)propyl] borate, -, -; bis(2-chloropropyl)
     1-(diisopropoxyphosphinyl)-2-methylpropyl borate, -, 1.4383;
    bis(2-bromopropyl) 1-(diethoxyphosphinyl)propyl borate, -, -;
    bis(2-chloropropyl) 1-[bis(2-chloroethoxy)phosphinyl]propyl borate, -, -;
     Bu 1-(diethoxyphosphinyl)propyl benzeneboronate, 98.4, 1.4780;
     3-diethoxyphosphinyl-3-pentyl diphenylborinate, 95, 1.5458;
    bis[1-(diethoxyphosphinyl)propyl] benzeneboronate, 97.7, 1.4870;
     .alpha.-diethoxyphosphinyl-4-methoxybenzyl diphenylborinate, 95, 1.5581;
     dibutyl .alpha.-bis(2-ethylhexyloxy)phosphinyl benzyl borate, 98, 1.4635;
     decyl bis[1-(diethoxyphosphinyl)propyl] borate, 97.6, 1.4373; dibutyl
     1-(diethoxyphosphinyl)propyl borate, 100, 1.4219; diallyl
     1-(dimethoxyphosphinyl)-2-trichloroethyl borate, -, 1.4782; diallyl
     1(ethoxyphenylphosphinyl)propyl borate, -, -; diallyl
     1(diphenylphosphinyl)propyl borate, -, 1.5680; S,S-dibutyl
     1-(diethoxyphosphinyl)propyl dithioborate, 90, -; S-ethyl
     bis[1-(diethoxyphosphinyl)propyl] thioborate, -, -; S,S-dibutyl
     1-(diethoxyphosphinyl)-2,2-dimethyl-4-cyanobutyl dithioborate, -, -;
     bis[1-(diethoxyphosphinyl)propoxy]dimethylaminoborane, 89, 1.4514;
     dipiperidino-2-[(diethoxyphosphinyl)propoxy]borane, -, 1.4926.
L4
    ANSWER 257 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN
AN
     1962:53482 CAPLUS
DN
     56:53482
OREF 56:10175b-q
     Organoboron compounds. Aromatic compounds
TI
ΑU
     Washburn, Robert M.; Billig, Franklin; Bloom, Murray; Albright, Charles
     F.; Levens, Ernest
CS
     Am. Potash and Chem. Corp., Whittier, CA
SO
     Advances in Chem. Ser. (1961), 32, 208-20
DT
     Journal
LΑ
     Unavailable
TΤ
     73774-44-4, Borinic acid, di-o-tolyl- 73774-45-5,
     Borinic acid, bis(p-methoxyphenyl)-
        (prepn. of)
RN
     73774-44-4 CAPLUS
CN
     Borinic acid, bis(2-methylphenyl) - (9CI) (CA INDEX NAME)
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RN 73774-45-5 CAPLUS
CN Borinic acid, bis(4-methoxyphenyl)- (9CI) (CA INDEX NAME)

The mechanism of the reaction between Grignard reagents and borate esters AΒ to form substituted boranes was investigated and found to go through an intermediate, RB(OR1)3MgBr (I). Substituents which increased the stability of I increased the yield of boronic acids with respect to borinic acids. Thus, using an incremental method of addn., the yields of boronic and borinic acid from methyl borate with p-chloro-, p-methoxy-, o-methyl- and phenylmagnesium bromide were: 82.0,-; 50.5, 14.1; 33.0, 18.1; and 75.5,-. The corresponding yields when PhMgBr was allowed to react with Et, Pr, iso-Pr, and Bu borate were: 33.0, 21.1; 14.1, 76.3; 16.1, 69.7; and 15.5, 77.6. When tetrahydrofuran was used as a solvent for the reaction of PhMgBr with (MeO)3B, the yield rose to 82.6%, while when pyridine was the solvent, the yield only rose to 78%. To check on the conditions under which B-C bonds were hydrolyzed the following expts. were performed. Benzeneboronic anhydride (0.58 mole) was heated with .beta.-ethoxyethanol (3.73 moles) for two hrs. while the water was removed as diisobutylene azeotrope. On distn. 83.3% tris(.beta.-ethoxyethyl) borate and 16.7% bis(.beta.-ethoxyethyl) benzeneboronate were obtained. The mixt. b. 93.degree./0.3 mm. and 0.434 mole C6H6 was isolated by vapor phase chromatography. Essentially similar results were obtained with starting ratios of 2:1 and 1:2. PhB(OH)2, (7.0 g.) was heated with BuOH (18.5 g.) 32 hrs. Vapor phase chromatography indicated 0.56% of the B-C bonds were cleaved; when tetrahydropyran was present 0.75% of the B-C bonds were broken; when H2O was removed as BuOH-H2O azeotrope 86% PhB(OBu)2, b. 104-6.degree./1.0 to 1.5 mm., was obtained; however, when the H2O was removed as a diisobutylene-H2O azeotrope, which took 156 hrs., 95% (BuO)3B was obtained. PhB(OH)2 (10.0 g.) and PhOH (22.0 g.) were heated together 2.5 hrs. while the H2O was removed as a diisobutylene-H2O azeotrope. Analysis showed 7% of the B-C bonds had been broken. When xylene was used as the azeotroping agent a mixt. of PhB(OPh)2 (77.1%) and (PhO)3B (23.9%), b. 155-9.degree./2 mm., m. 113-15.degree., was obtained. Very pure PhB(OH)2 was heated in an inert atm. with MeOH 6 days at which time 0.069% of the B-C bonds had been cleaved; similarly with acetone 0.13% of the B-C bonds were cleaved. Very pure PhB(OH)2 was dissolved in basic soln. for 8 hrs.; no C6H6 could be found. After slow acidification no trace of C6H6 could be found and all the starting material was recovered. However, in the presence of MeOH less than 1% of the B-C bonds were cleaved in acid soln., while in the presence of pyridine almost 20% of the B-C bonds were broken.

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L4 ANSWER 258 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN
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AN 1961:99427 CAPLUS

DN 55:99427

OREF 55:18712f-i,18713a-i,18714a-f

TI 5-Nitro-2-(furyl-substituted) 1,3,4-oxadiazoles, 1,3,4-thiadiazoles, and 1,3,5-triazines

AU Sherman, William R.

CS Abbott Labs., N. Chicago

SO Journal of Organic Chemistry (1961), 26, 88-95

CODEN: JOCEAH; ISSN: 0022-3263

DT Journal

LA Unavailable

GI For diagram(s), see printed CA Issue.

AB New types of antibacterial nitrofurans, 1,3,4-oxadiazoles, and 1,3,4-thiadiazoles contg. the system C:N.N.C: in a heterocycle, and 1,3,5-triazines lacking such a system were prepd. CNBr (48.0 g.) and 68.4 q. 5-nitro-2-furoylhydrazine (I) refluxed 1 hr. in 2 l. MeOH, the cooled mixt. filtered from 20.4 g. product, the mother liquor concd., the brown oil poured into 500 ml. H2O to give 30 g. product, the 2 crops combined, and crystd. from HCONMe2-alc. gave 64% 2-amino-5-(5-nitro-2-furyl)-1,3,4oxadiazole (II), m. 258-60.degree. (decompn.). Pb304 (34.2 g.) and 4.60 q. 1-(5-nitro-2-furoyl)thiosemicarbazide refluxed 24 hrs. in 250 ml. alc., filtered, and the residue extd. with hot alc. yielded 16% II. I (50.15 g.) in 500 ml. 10:1 H2O-HCl stirred 1 hr. (ice bath) with introduction of COC12 below the surface and the H2O-washed product (92%, m. 200-2.degree.) crystd. from Me2CO-H2O gave 5-(5-nitro-2-furyl)-1,3,4-oxadiazol-2-one (III), m. 201-2.degree. (decompn.), .lambda. 5.64 .mu. (Nujol), supporting the oxo structure; N-Ac deriv. m. 144-4.5.degree., .lambda. 5.50, 5.70 .mu. (CHCl3). I (17.11 g.) in 375 ml. dioxane satd. with CSCl2 at room temp., the mixt. treated with C, and the filtered soln. dild. with 700 ml. C6H14 yielded 65% 5-(5-nitro-2-furyl)-1,3,4-oxadiazole-2-thione (IV), m. 157.5-8.degree. (decompn.). III (9.85 g.) and 3.75 ml. HCHO boiled in 40 ml. H2O and kept 30 min. on a steam bath gave 4.7% 3-hydroxymethyl-5-(5nitro-2-furyl)-1,3,4-oxadiazol-2-one, m. 110-11.degree. (alc.-C6H12). Several 3-aminomethyl oxadiazolones (V) were readily obtained by the Mannich reaction. III (19.7 g.) in 200 ml. ice-cold abs. alc. stirred 10 min. with addn. of 7.5 ml. HCHO and 30 ml. alc. contg. 4.5 g. HNMe2, the ppt. crystd. from alc., and the product (14 g., m. 117-47.degree.) extd. with 800 ml. hot C6H6 yielded starting material, unidentified product, and 28% V (R = Me) (VI), m. 118-19.degree.. III (19.71 g.) in 200 ml. hot abs. alc. contg. 10 ml. HCONMe2 cooled to 40.degree., treated with 7.5 ml. HCHO and 7.31 g. HNEt2, and the mixt. cooled (ice bath) yielded 22% V (R =Et) (VII), m. 99-9.5.degree.. The procedure used for VI converted 8.71 g. morpholine into 27.4 g. V (NR2 = morpholino), m. 190-1.degree. (HCONMe2-alc.). The general procedure for VII gave V (NR2 = piperidino, pyrrolidino, and hexamethylenimino), m. 133.5-4.degree. (alc.), 138-9.degree. (HCONMe2-alc.), and 134-5.degree., in 78.2, 85, and 92% yields, resp. With 4.31 g. piperazine, the Mannich procedure converted III immediately to 23.9 g. orange 1,4-bis[5-(5-nitro-2-furyl)-2-oxo-1,3,4oxadiazol-3-ylmethyl]piperazine, m. 200.degree. (decompn.). The Mannich base (0.003 mole) in 10 ml. H2O stirred with 0.003 mole concd. HCl gave 94% III. IV (6.39 g.) in 30 ml. alc. treated with 6 ml. MeI and 75 ml. alc. contg. 1.68 g. KOH yielded 85% material, m. 164.5-5.degree., crystd. from HCONMe2-H2O to give 2-methylthio-5-(5-nitro-2-furyl)-1,3,4-oxadiazole (VIII), m. 165.5-6.degree., with 93% recovery. Treatment of IV with HgO in either refluxing H2O or dioxane gave only the Hq bis salt, C12H4HgN6O8S2, m. 262.degree. (decompn.) (HCONMe2-H2O), and not the expected III. VIII (0.90 g.) heated 7 hrs. on a steam bath in 20 ml. concd. HCl with evolution of MeHS and the soln. cooled gave 74% 5-nitro-2

furoic acid, m. 182-3.degree. (decompn.). The most active member in this series was III. III and the acid-labile Mannich derivs. were effective against gram-pos. and gram-neg. infections in animals by intramuscular and oral routes. IV had no in vitro or in vivo activity. PhNHNH2 (4.32 g.) in 25 ml. ice-cold dry C6H6 treated dropwise with 3.51 g. 5-nitro-2-furoyl chloride (IX) in 25 ml. dry C6H6, the mixt. refluxed several min., the cooled mixt. filtered from 37.8% 1-(5-nitro-2-furoyl)-1-phenylhydrazine (X), m. 167-7.5.degree. (abs. alc.), the filtrate evapd., the residue extd. with warm H2O, and crystd. from PhMe yielded 17.4% 1-(5-nitro-2-furoy1)-2-phenylhydrazine (XI), m. 127.5-8.degree.. g.) in 50 ml. dry Et2O added slowly with stirring to 4.32 g. PhNHNH2 in 50 ml. dry Et2O, the residue on filtration washed with H2O, taken up in hot alc., the soln. cooled, filtered from 0.80 g. X, the mother liquor and the Et20 filtrate combined, and the solvents evapd. yielded 58.2% XI, m. 124.5-5.5.degree.. XI (1.80 g.) in 50 ml. PhMe heated on a steam bath 1 hr. with passage of COCl2, the solvent evapd., and the residue slurried in 25 ml. boiling alc. gave 84% 5-(5-nitro-2-furyl)-3-phenyl-1,3,4-oxadiazol-2-one (XII), m. 186.5-7.5.degree. (PhMe). X (0.5 g.) heated 40 min. on a steam bath with 5 ml. freshly distd. BzH gave 78% benzylidene deriv., m. 207-8.degree. (PhMe). XII was physiol. inactive. IX (5.27 q.) in 30 ml. pure dry dioxane added slowly with stirring to 2.73 g. H2NCSNHNH2 and 7 g. NaHCO3 in 50 ml. dry dioxane, the mixt. stirred 2 hrs. at 20.degree. and 10 min. at 100.degree., the cooled filtered soln. concd., and dild. with alc. yielded 46% 5,2-O2NC4H2OCONHNHCSNHR (XIII, R = H) (XIV), m. 192.degree. (decompn.) (alc.), also prepd. in 50% yield by heating 34.22 g. 5-nitro-2-furoylhydrazine (XV), 25 g. KCNS, and 20 ml. concd. HCl 4 hrs. in 300 ml. H2O on a steam bath, cooling overnight, filtering, and slurrying the ppt. in a small vol. of boiling alc. several min. XV (17.11 g.) and 8.04 g. MeNCS refluxed 2 hrs. in 250 ml. alc. and the cooled mixt. filtered gave XIII (R = Me).EtOH m. 166.5-7.degree. (alc.), converted by heating in vacuo at 100.degree. to XIII (R = Me) (XVI), m. 190.degree. (decompn.). Similarly were prepd. the corresponding XIII (R = Et, Ph) (XVII, XVIII), m. 193.degree. (alc.), and 175.degree. (decompn.) (Me2CO-H2O), in 88 and 81% yields, resp. XIV (11.51 g.) added portionwise with stirring to concd. H2SO4 at 0.degree., the mixt. stirred 1 hr. at 0.degree. and 1 hr. at 0-20.degree., the filtered soln. poured over cracked ice, kept 16 hrs. at 0.degree., filtered, the acid soln. neutralized, the products combined, and recrystd. from HCONMe2-H2O gave 76% 2-amino-5-(5-nitro-2-furyl)-1,3,4-thiadiazole (XIX, R = H) (XX), m. 280.degree. (decompn.). XX (7.0 g.) and 8 ml. Ac20 kept 2 hrs. at 80-90.degree. in 200 ml. C5H5N, cooled, and the product crystd. from C5H5N yielded 95% XIX (R = Ac), m. 308.degree. (decompn.). Similar cyclization of XVI, XVII, and XVIII yielded the corresponding XIX (R = Me, Et, and Ph), m. 214.5-15.5.degree. (HCONMe2-H2O), 216-16.5.degree. (HCONMe2-H2O), and 267-7.5.degree. (HCONMe2-H2O), in 52, 70 and 73% yields, resp. XIV was the most active thiadiazole antibacterial, the substituted amino derivs. showing decreasing activity in the order R = Me, Et, Ph. MeOCH2CH2OH (50 ml.) contg. 1.15 g. Na treated with 8.98 g. H2NC(:NH)NHC(:NH)NHR (XXI, R = iso-Pr), the filtered soln. kept 3 days at 20.degree. with 8.55 g. Me 5nitro-2-furoate (XXI, R = iso-Pr), the filtered soln. kept 3 days at 20.degree. with 8.55 g. Me 5-nitro-2-furoate (XXII), filtered from 2.73 g. alc.-washed product, m. 192.5-4.degree., the filtrate kept 2 weeks before evapn., and the residue extd. with hot alc. gave 0.93 g. product, crystd. (total yield 28%) from alc. to give the 1,3,5-triazine (XXIII) (R = Me2CH, R' = H), m. 197-7.5.degree.. The corresponding XXIII (R' = H, R = Ph, o-MeC6H4, p-ClC6H4, p-O2NC6H4), m. 259.5-60.degree. (BuOH), 257.degree. (decompn.) (BuOH), 249-9.5.degree. (HCONMe2-H2O), and 342.degree. (decompn.) (HCONMe2), were similarly

obtained in 15.4, 9.6, 8.5, and 5% yield, resp. In an attempt to prep. XXIII (R' = H, R = o-MeC6H4) with IX and XXI (R = o-MeC6H4), no cyclized product was obtained. The reduced rate of cyclization may have resulted from a combination of steric and electric effects and long standing might destroy large amts. of the base-labile nitrofuran intermediates or products. XXIII (R = R' = H) (XXIV) was formed in 80% yield after reaction of XXII with XXI (R = H) in 16 hrs. at 20.degree.. XXIV had a high degree of antibacterial activity in animals by the intramuscular route. XXIV refluxed with Ac2O gave the di-Ac deriv., XXIII (R = R' = Ac), m. 274.5-5.degree. (decompn.) (HCONMe2), converted by refluxing 16 hrs. in 1:1 HCONMe2-H2O to the mono-Ac deriv., XXIII (R = Ac, R' = H), m. 317.degree. (decompn.) (HCONMe2). XXII (8.55 g.) and 11.10 g. XXI (R =p-O2NC6H4) kept 14 days at 20.degree. in 200 ml. MeOCH2CH2OH yielded 56% yellow solid, m. 252.degree. (decompn.), crystd. from AcOH-alc. to give 1-(5-nitro-2-furyl)-5-(p-nitrophenyl)biguanide-H2O, m. 259.degree., converted by introduction (6.75 g.) into a few ml. of boiling HCONMe2 and immediate cooling to yield 33% XXIII (R' = H, R = p-O2NC6H4) (XXV), m. 338.degree. (decompn.), and 4.35 g. starting material, m. 245.degree. (decompn.). XXII (8.6 g.) and XXI (R = p-O2NC6H4) heated 8.5 hrs. on a steam bath in 50 ml. HCONMe2 and the soln. cooled yielded 12% XXV. The immediate formation of XXV without isolation of the biguanide intermediate was due to the greater soly. of the intermediate in HCONMe2, facilitating cyclization.

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ANSWER 259 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN
L4
AN
     1961:76160 CAPLUS
DN
     55:76160
OREF 55:14457b-d
     Coordination compounds from diphenylboric acid and substances with ring
     nitrogen as the electron donors
ΑU
     Neu, R.
CS
     Dr. Willmar Schwabe G. m. b. H., Karlsruhe, Germany
SO
    Arch. Pharm. (1961), 294, 173-8
DT
     Journal
LΑ
     Unavailable
ΙT
     2622-89-1, Borinic acid, diphenyl-
        (prepn. of)
RN
     2622-89-1 CAPLUS
     Borinic acid, diphenyl- (6CI, 7CI, 8CI, 9CI)
CN
                                                   (CA INDEX NAME)
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Ph
|
|
| Ph— B— OH
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AB Pyridine-2-carbinol (371 mg.) in 10 ml. C6H6 was mixed with 580 mg. Ph2BOH in 80 ml. petr. ether to give 84.3% of the O,O-diphenylboryl ester, m. 147.degree. (heptane). Similarly were prepd. the corresponding O,O-diphenyl or O,O'-bis(diphenylboryl) esters from the following aromatic amines (amine and m.p. and % yield of boron compd. given): 6-methylpyridine-2-carbinol, 184-6.degree., 71.4; quinoline-2-carbinol, 185-6.degree., 68.4; bis(2-pyridyl)-glycol, 279-81.degree., 100; 2,2'-pyridoin (I),-, 94.8; 6,6'-di-Me deriv. of I, 281-3.degree., 87.3; pyridine-2-ethanol, 166-9.degree., 85.4.

L4 ANSWER 260 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN AN 1961:64849 CAPLUS

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DN
     55:64849
OREF 55:12328a-b
ΤI
     Action of salts of trivalent metals and non-metals on sodium
     tetraphenylboron
ΑU
     Neu, R.
CS
     Chem. Lab. der Fa. Dr. Willmar Schwabe G. m. b. H., Karlsruhe-Durlach,
     Germany
SO
     Arch. Pharm. (1961), 294, 7-10
DT
     Journal
LΑ
     Unavailable
IT
     2622-89-1, Borinic acid, diphenyl-
        (formation in NaBPh4 reaction with salts)
RN
     2622-89-1 CAPLUS
     Borinic acid, diphenyl- (6CI, 7CI, 8CI, 9CI) (CA INDEX NAME)
CN
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Page 335

Ph | Ph— B— OH

AB NaBPh4 (0.01 mole) in 100 ml. H2O was treated with 0.0003 mole salt in 10 ml. H2O, the soln. steam-distd., and the distillate contg. Ph2BOH (I) treated with 3 g. 8-hydroxyquinoline in CHCl3, MeOH, or in H2O contg. H2NCH2CH2OH (salt and % yield I given): AlCl3.6H2O, 71; TiCl3, 81; FeCl3.6H2O, 84; CrCl6, 30; Bi(NO3)3.5H2O, 36; SbCl3, 72.

ANSWER 261 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN L4AN 1961:50776 CAPLUS DN 55:50776 OREF 55:9789a-b TΙ Colorimetric determination of tetracycline with diphenylborinic acid AU Uno, Toyozo; Morishita, Nobumichi CS Univ. Kyoto SO Yakugaku Zasshi (1960), 80, 1679-81 CODEN: YKKZAJ; ISSN: 0031-6903 DTJournal

CODEN: YKKZAJ; ISSN: 0031-6903
DT Journal
LA Unavailable
IT 2622-89-1, Borinic acid, diphenyl-

(in detn. of chlortetracycline, oxytetracycline and tetracycline)

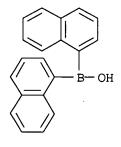
RN 2622-89-1 CAPLUS

CN Borinic acid, diphenyl- (6CI, 7CI, 8CI, 9CI) (CA INDEX NAME)

Ph | Ph— B— OH

AB To 5 ml. aq. soln. contg. 10-30 .gamma. tetracycline (I), 5 ml. 1% Ph2BOH in EtOH is added and the soln. dild. (to compensate for vol. contraction) to 10 ml. with 99% EtOH. Optical d. at 415 m.mu. is measured after 1 hr. The blank test is carried out with 5 ml. H2O, 5 ml. of the reagent and the soln. is made up to 10 ml. with 99% EtOH. Chlor- and oxytetracyclines can be detd. in a similar manner, using the absorption max. at 417 and 407 m.mu., resp. The values obtained by this method and biol. assay on aq. solns. of I allowed to stand for a long period of time were in good agreement.

L4 ANSWER 262 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN ΑN 1961:48583 CAPLUS DN 55:48583 OREF 55:9353c-f Organoboron compounds. LVIII. Action of amines and ammonia on diarylboron chlorides ΑU Mikhailov, B. M.; Fedotov, N. S. CS N. D. Zelinskii Inst. Org. Chem., Moscow SO Izvestiya Akademii Nauk SSSR, Seriya Khimicheskaya (1960) 1590-4 CODEN: IASKA6; ISSN: 0002-3353 DT Journal LΑ Unavailable IT62981-91-3, Borinic acid, di-1-naphthyl-(prepn. of) RN 62981-91-3 CAPLUS CN Borinic acid, di-1-naphthalenyl- (9CI) (CA INDEX NAME)



AB cf. CA 55, 360c. Spontaneous reaction of Ph2BCl with 2 moles PhNH2 in Et20 gave 76.2% Ph2BNHPh b0.04 134-5.degree., m. 57-9.degree. (sealed tube), hydrolyzed by moist air. Treatment of Ph2BCl in isopentane with dry NH3 at 0.degree. gave 87.2% Ph2BCl.2NH3 (softened at 175-80.degree. but did not m. even at 250.degree.). Ph2BCl and Et3N in heptane gave Ph2BCl.Et3N, m. 125-33.degree.. Passage of NH3 into refluxing soln. of Ph2BCl in C6H6 gave 71.7% Ph3B.NH3, m. 216-17.degree.. Since Ph2BCl.2NH3 treated with H2O was converted into 69.3% [Ph2B(OH)2]NH4, decompg. at 106-8.degree., it appeared that the original complex was indeed [Ph2B(NH2)Cl]NH4. Reaction of (1-Cl0H7)2BCl with MeNH2 in C6H6 at room temp. gave 81% (1-C10H7)2BNHMe, m. 104-6.degree., slowly hydrolyzed by moist air; heated with H2O, this gave C10H8 and .alpha.-naphthylboronic acid, m. 200-3.degree.. Similarly was prepd. 81.4% (1-C10H7)2BNHPh, m. 125-7.degree., rapidly hydrolyzed by moist air to yield di-alpha.-naphthylboronic acid, m. 111-14.degree.. Similarly, iso-BuNH2 gave 80.6% viscous (1-C10H7)2BNHCH2CHME2, b1 240-5.degree., hydrolyzed only by hot H2O to .alpha.-naphthylboronic acid, C10H8, and iso-BuNH2. Use of NH3 in the above reaction gave 87% (1-C10H7)2BNH2, m. 113 14.degree., oxidized by air and hydrolyzed by hot H2O to C10H8 and .alpha.-naphthylboronic acid. Et2NH similarly gave 77.6% (1-C10H7)2BNEt2, m. 178-9.degree., resistant to air oxidn. and hydrolysis. Heating (1-C10H7)2BOH with H2O 1 hr. gave C10H8 and 1-C10H7B(OH)2, m. 208-9.degree..

L4 ANSWER 263 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

AN 1961:48546 CAPLUS

DN 55:48546 OREF 55:9345e-g

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10085368.2
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Page 337

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TI
    Diarylboric acids and their anhydrides
ΙN
    Neu, Richard; Buechl, Hermann
PA
    Heyl & Co. Chemisch-Pharmazeutische Fabrik
DT
    Patent
LΑ
    Unavailable
FAN.CNT 1
    PATENT NO.
                  KIND DATE
                                      APPLICATION NO.
                                                      DATE
    -----
                                       ______
                                                      _____
PΙ
    DE 1040550
                         19581009
IT
    2622-89-1, Borinic acid, diphenyl-
       (prepn. of)
RN
    2622-89-1 CAPLUS
CN
    Borinic acid, diphenyl- (6CI, 7CI, 8CI, 9CI) (CA INDEX NAME)
```

Ph | | | Ph— B— OH

T.4

Diarylboric acids are prepd. by interaction of B trihalides, such as BF3, BCl3, BF3.Et2O, boric acid esters, preferably alkyl esters, and monoarylboric acid esters, such as phenylboric acid alkyl esters, with arylmagnesium halides in Et2O, acid hydrolysis of the arylboron compds. formed under mild conditions, e.g. in the cold, and isolation of the products. Thus, a Grignard soln. (10 g. Mg and 65 g. PhBr) is dropped into 15 g. BF3.Et2O in 80 cc. Et2O with stirring and refluxing, the mixt. poured into 16 g. NaOH, 100 cc. H2O, and 200 g. crushed ice, 2N HCl added, the layers sepd., the aq. layer extd. several times with Et2O, the Et2O exts. evapd., the residue steam distd., the distillate extd. with Et2O, the ether layer dried, and evapd. to give 6 g. oily diphenylboric acid, which solidifies gradually and yields tetraphenyldiboron oxide after recrystn. from petr. ether, m. 118-20.degree.. In the same manner is prepd. tetra-p-tolyldiboron oxide, m. 105-6.degree.

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AN
    1961:40246 CAPLUS
DN
    55:40246
OREF 55:7826e-q
TI
    Preventing icing in jet fuels
    Steinberg, Howard; Hunter, Don L.; Goda, Ernest H.
PA
    United States Borax & Chemical Corp.
DT
    Patent
T<sub>2</sub>A
    Unavailable
FAN.CNT 1
    PATENT NO.
                   KIND DATE
                                        APPLICATION NO. DATE
    -----
                                        -----
                                                        -----
PI
    US 2960819
                          19601122
                                        US
    2622-89-1, Borinic acid, diphenyl-
IT
       (and derivs., esters, icing prevention in jet fuel by)
RN
    2622-89-1 CAPLUS
```

Borinic acid, diphenyl- (6CI, 7CI, 8CI, 9CI) (CA INDEX NAME)

ANSWER 264 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

Ph | Ph— B— OH

AB To prevent icing in kerosine jet-engine fuel (JP-4) down to the freezing temp. of the fuel itself, 0.01-1.0% by wt. of a H2O-unstable ester of boric acid, a benzeneboronic acid, an alkaneboronic acid, a diphenylborinic acid, or a dialkylborinic acid is added. The esters are derived from alcs. having 1-12 C atoms or from H2O-unstable biborates or boric anhydrides of 1,2- and 1,3-glycols. The following additives are mentioned in the claims: tris(hexylene glycol) biborate, bis(hexylene glycol) boric anhydride, tris(2-ethylhexyl) borate, Et3BO3, or diisopropyl benzeneboronate. For example, 0.1% Et3BO3 prevents ice formation down to -60.degree.

L4ANSWER 265 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN AN 1961:35817 CAPLUS DN 55:35817 OREF 55:6970b-d Physical properties of organoboron compounds TIΑU Christopher, Phoebus M. Newark Coll. of Eng., Newark, NJ CS SO Journal of Chemical and Engineering Data (1960), 5, 568-70 CODEN: JCEAAX; ISSN: 0021-9568 DTJournal Unavailable LΑ 2622-89-1, Borinic acid, diphenyl-ΙT (alkyl esters, phys. property equations for) RN2622-89-1 CAPLUS

Borinic acid, diphenyl- (6CI, 7CI, 8CI, 9CI) (CA INDEX NAME)

CN

Equations are given for the molar refractions (R) and vols. of the following organoboron compds. as a function of the number of C atoms in the alkyl substituents: B(OR)3, ClB(OR)2, ROBCl2, BR3, PhB(OR)2, MeC6H4B(OR)2, Ph2BOR, B[O(CH2)nCl]3, ClB [O(CH2)nCl]2, Cl(CH2)nOBCl2, PhB(OR)Cl, C6H4O2BOR, (ROBO)3, (RBO)3, [B(NHR)NR]3, B(NHR)3. The equations are Robs. = an + b and Vtm = cn + d, where n is the number of carbons per R group (1 to 6), and a, b, c, d are consts. The molar refraction and vol., normal b.p., enthalpy of vaporization, entropy of boiling, internal pressure, crit. pressure, vol. and temp., crit. ratio (Tc/Tb), van der Waals correction factors, and mol. radius were calcd. for Pr3B, Bu3B, and 14 B(OR)3 compds., where R = Et, Pr, Bu, iso-Bu, sec-Bu, tert-Bu, n-amyl, isoamyl, iso-PrMeCH, PrMeCH, n-octyl, CH3(CH2)5Me2CH, CH2:CHCH2, Cl(CH2)2, and (ClCH2)2CH.

L4 ANSWER 266 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN
AN 1961:33054 CAPLUS
DN 55:33054
OREF 55:6472g-i,6473a-f
TI Arylboronic acids. V. Methyl-substituted boronic acids, borinic acids, and

triarylborons

H. Hawking Dishard T. Lauren William J. Godin W. B.

AU Hawkins, Richard T.; Lennarz, William J.; Snyder, H. R.

CS Univ. of Illinois, Urbana

SO Journal of the American Chemical Society (1960), 82, 3053-9 CODEN: JACSAT; ISSN: 0002-7863

DT Journal

LA Unavailable

IT 20631-84-9, Borinic acid, dimesityl-

(prepn. of)

RN 20631-84-9 CAPLUS

CN Borinic acid, bis(2,4,6-trimethylphenyl) - (9CI) (CA INDEX NAME)

cf. CA 53, 2131c. To 10 g. Mg turnings heated to 90.degree. was added . AB slowly, with stirring under N, a soln. of 59.67 g. mesityl bromide in 60 ml. anhyd. tetrahydrofuran. Reaction started within 2 min.; the rate of addn. of the halide was adjusted to maintain the temp. at 90.degree... reaction temp. was kept at 95.degree. for 2 hrs. The cooled mixt. was dild. to 120 ml. with tetrahydrofuran (96% yield). A soln. of 0.275 mole 2,4,6-Me3C6H2MgBr and a soln. of 34.0 ml. (MeO)3B were added simultaneously to 200 ml. of vigorously stirred dry ether at -60 to -70.degree.. After a half hr. at -70.degree., the mixt. was allowed to warm to 0.degree., stirred 1 hr., and hydrolyzed by the addn. of 100 ml. H2O (overnight). The org. layer was sepd. from the aq. layer and the latter extd. three times with Et2O. The ether phase was washed twice with H2O, thrice with dil. HCl, and twice more with H2O. The ether was removed by dropping the soln. into a warmed flask with stirrer. As the head temp. approached 42.degree., 650 ml H2O was added slowly; 90 ml. H2O was collected while the temp. rose to 103.degree.. By cooling, filtering, concg., and filtering again, 35.5 g. (72%) mesityleneboronic acid (I) was obtained. Upon heating, loss of H2O was observed as low as 80.degree.. Pure I was dried in a vacuum desiccator for several days, recrystd. from pet. ether, and sublimed at 170.degree./1-2 mm. to give a dimer, m. 220-2.degree.. Dimesityleneborinic acid, m. 142.degree., was prepd. by hydrolysis of (2,4,6-Me3C6H2)2BF (94.5% yield). Trimesitylboron (II) was prepd. in the usual manner. The crude product, which sepd. upon addn. of 95% EtOH, was recrystd. twice from EtOAc and then sublimed at 140.degree. and 2 mm., m. 195-7.degree.. Upon addn. of EtOH, a small crop of crystals was obtained, which were recrystd. (petr. ether-Me2CO, and then petr. ether). Sublimation at 230.degree. and 0.2 mm. yielded [(2,4,6-Me3C6H2)2B]2O. The material was polymorphic (the more stable modification showed the lower m.p.), m. 267-8.degree. and 289-90.degree.. To a mixt. of 7.0 ml. white fuming HNO3 and 4.0 ml. concd. H2SO4 was added (10 min.) 500 mg. II. The thick brown slurry was stirred 10 min. at -70.degree., allowed to warm to -40.degree., and dild. with 15 ml. cold H2O. Recrystn. from EtOAc and drying 6 hrs. at 165.degree. and 0.8 mm. gave 450 mg. (51.3%) tris(3,5-dinitro-2,4,6-trimethylphenyl)boron (III). III did not melt up to 350.degree., but started to decomp. at 290.degree.. A mixt. of 3.28 g. I and 2.16 g. o-(H2N)2C6H4 in 25 ml. MePh was heated until all the H2O was removed. The product was concd. in vacuo, dild. with 20 ml. petr. ether, and filtered. Unreacted o-(H2N)2C6H4 was sublimed (5 hrs.) at 100.degree. and 0.5 mm. The residue was dissolved in 80 ml. warm C6H6, filtered, concd., and cooled. 2-Mesityl-1,3-dihydro-2,1,3-benzoboradiazole, m. 141.degree., (2.18 g., 46.4%) was obtained. 2,6-Dimethylbenzeneboronic acid (IV), m. 125-30.degree., was prepd. in essentially the same manner as I. 2,6-Dimethylboronic anhydride (V), m.

144-7.degree., was prepared by the two-fold sublimation of IV at 125.degree. (0.05 mm.). V (300 mg.) was treated with a soln. of 56.8 g. pyridine in 5.0 ml. abs. Et20. The anhydride appeared to dissolve and new crystals formed. The crystals were washed with ether and with petr. ether to give 271.7 mg. (76%) pyridine-V complex, m. 170-5.degree.. A soln. of 4.00 g. V in 40 ml. CCl4 was treated with 1.78 g. recrystd. N-bromosuccinimide (VI) and 8.3 mg. Bz2O2. The mixt. was irradiated (200 watt lamp) and refluxed. After 15 min., 3.56 g. VI and 16.6 mg. Bz202 were added and irradiation and refluxing were continued 1.5 hrs. Succinimide (2.87 g., 87%) and 6.29 g. (99%) 2-bromomethyl-6methylbenzeneboronic anhydride (VII) were isolated. VII was added to a stirred mixt. of tetrahydrofuran and a soln. of 1.43 g. NaOH in 25 ml. H2O. The mixt. was stirred overnight, the pH adjusted to 2-3, and the aq. layer extd. (Et20). The combined Et20 washings and tetrahydrofuran layer were concd. in vacuo. The residue was extd. with 130 ml. boiling 10% EtOH. After two days, crystn. occurred. The product was sublimed 20 hrs. at 45.degree. (0.05 mm.) to yield 330 mg. 6-methylboronophthalide, m. 115-25.degree..

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L4
     ANSWER 267 OF 309 CAPLUS COPYRIGHT 2003
                                                 ` on STN
AN
     1961:13146 CAPLUS
DN
     55:13146
OREF 55:2531a-b
TI
     Simple method of preparation of dipheny
ΑU
     Neu, Richard
CS
     Willmar Schwabe, Karlsruhe-Durlach, Ge
SO
     Naturwissenschaften (1960), 47, 304
     CODEN: NATWAY; ISSN: 0028-1042
DT
     Journal
LΑ
     Unavailable
IT
     2622-89-1, Borinic acid, diphenyl-
        (prepn. of)
RN
     2622-89-1 CAPLUS
CN
     Borinic acid, diphenyl- (6CI, 7CI, 8CI, 9CI)
                                                   (CA INDEX N.
```

Ph | Ph— B— OH

AB After a review on the known prepn. methods of Ph2BOH (I), the author proposed a convenient method for obtaining larger quantities. By influence of salts of tervalent metals or metalloids on NaBPh4 in aq. soln. and by steam distn. I was obtained and could be isolated as the anhydride (60% yield) or as the quinoline complex (84% yield).

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L4 ANSWER 268 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN
AN 1961:6906 CAPLUS
DN 55:6906
OREF 55:1292e-f
TI The identification of boron in organic bondings
AU Neu, Richard
CS Chemisch Forschungslaboratorium der Fa. Dr. Willmar Schwabe G. m. b. H.,
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Karlsruhe-Durlach

SO Zeitschrift fuer Analytische Chemie (1960), 176, 343-6

CODEN: ZANCA8; ISSN: 0372-7920
DT Journal

LA Unavailable

RN 2622-89-1 CAPLUS

CN Borinic acid, diphenyl- (6CI, 7CI, 8CI, 9CI) (CA INDEX NAME)

(esters, color reactions with diphenylcarbazone

AB Diphenylcarbazone gives a blue-violet color with B bonded to 1 or 2 C atoms, as in the aromatic boric acids. In the presence of the esters of these acids, a red-violet or red color results. N-contg. boric acid esters (such as triethanolamine borate) give no reaction. The Zeiss spectrophotometer shows a max. at 551 m.mu. for phenylboric acid, and at 571 m.mu. for diphenylboric acid. Similar results were obtained with diphenylcarbazide as identifying agent for diphenylboric acid anhydride. 22 references.

L4 ANSWER 269 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

AN 1960:121117 CAPLUS

DN 54:121117

OREF 54:23168f-g

TI Nematocides

IN Jacobi, Ernst; Lust, Siegmund; Zima, Otto; van Schoor, Albert

SO From: C.Z. 1959, 8294..

DT Patent

LA Unavailable

FAN.CNT 1

PATENT NO. KIND DATE APPLICATION NO. DATE

DΕ

PI DE 1044500 19581120

IT 89566-59-6, Borinic acid, bis(p-chlorophenyl)-

(and derivs., nematocides)

RN 89566-59-6 CAPLUS

CN Borinic acid, bis(4-chlorophenyl) - (9CI) (CA INDEX NAME)

AB Compds. of the general formula (4-ClC6H4)2BOR are used, in which R is H, a univalent org. residue, a univalent metal atom, or an equiv. of a multivalent metal atom. The compds., e.g. bis(p-chlorophenyl)borinic acid, or its salts or esters, are obtained by reaction of Grignard compds. of p-chlorobromobenzene with the desired borate. R and b. p. given: isoBu, b0.2 158-60.degree.; Ph, b0.1 170-2.degree. 2-chloroethyl, b0.02 165-70.degree.; and allyl, b0.001 171-3.degree..

L4 ANSWER 270 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

9/24/2003>

AN 1960:88097 CAPLUS

DN 54:88097

OREF 54:16733e-g

TI Organic boron compounds as herbicides

IN Barnsley, Geoffrey E.; Eaton, John K.; Airs, Raymond S.

PA "Shell" Research Ltd.

DT Patent

LA Unavailable

FAN.CNT 1

PATENT NO. KIND DATE APPLICATION NO. DATE

PI DE 1016978 19571003 DE

IT 89566-59-6, Borinic acid, bis(p-chlorophenyl)-

(herbicide)

RN 89566-59-6 CAPLUS

CN Borinic acid, bis(4-chlorophenyl) - (9CI) (CA INDEX NAME)

Certain org. B compds. are selective herbicides and inhibitors for the growth of plants. Suitable compds. are benzeneboronic or diphenylborinic acids which are substituted by halogen, Me, halomethyl or methoxy; diphenylborinic acid; an anhydride, ester, salt or N complex of the monoor dibasic boric acid; and an anhydride, ester, or N complex of benzenaboronic acid. These B compds. are used with surface-active agents and (or) carriers. Examples are given showing effectiveness of 4-chloroor 4-bromo- and 4-methoxybenzeneboronic acid and bis(4-chlorophenyl)-borinic acid.

L4 ANSWER 271 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

AN 1960:88037 CAPLUS

DN 54:88037

OREF 54:16725e-g

TI Assessment of Na diphenylborinate as a selectiv

AU Barnsley, G. E.; Yates, J.

SO Proc. Brit. Weed Control Conf., 4th, Brighton 245-50

DT Journal

LA Unavailable

RN 95493-42-8 CAPLUS

CN Borinic acid, diphenyl-, sodium salt (6CI, 7CI) (CA INDEX NAME)

Na

AB The toxicity was studied of Na diphenylborinate (I) to a range of crops and annual weeds on the basis of seed mortality or growth inhibition. Tolerant crops to spray of I were cereals, grasses, onions, linseed, certain legumes, conifers, wheats, oats, barley, and certain varieties of peas. I induced both acute and chronic phytotoxicity, effects which were thought to be related to the decompn. products of I. The L.D.50 for rats of I was 400 mg./kg., orally. The introduction of Cl atoms into the phenyl nuclei of I resulted in loss of selectivity.

ANSWER 272 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN L4 AN 1960:85490 CAPLUS DN 54:85490 OREF 54:16256f-h TТ The titration of hydrazine sulfate with permanganate ΑU Drotschmann, C.; Wyatt, R. Chemisch Weekblad (1960), 56, 265-6 SO CODEN: CHWEAP; ISSN: 0009-2932 DTJournal LΑ Unavailable 2622-89-1, Borinic acid, diphenyl-IT (detn. of) RN 2622-89-1 CAPLUS

Borinic acid, diphenyl- (6CI, 7CI, 8CI, 9CI) (CA INDEX NAME)

CN

AB Dissolve 0.07 g. N2H4.H2SO4 (I) in 200 ml. water, and add 10 ml. H2SO4 (1: 4). Heat the soln. to about 80.degree., and titrate with 0.02N KMnO4 till the pink color changes to purple. The titration may also be done potentiometrically. The specific properties of a MnO2 sample det. the reaction velocity in the oxidn. of I by MnO2, an exothermic reaction. The velocity is measured by means of a temp.-time curve.

L4 ANSWER 273 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN AN 1960:85489 CAPLUS DN 54:85489 OREF 54:16256e-f

TI Mercurimetric titration of aromatic boron compounds. Reaction of aromatic boron derivatives with mercuric ion

AU Heyrovsky, Antonin CS Karlova Univ., Prague

SO Zeitschrift fuer Analytische Chemie (1960), 173, 301-9 CODEN: ZANCA8; ISSN: 0372-7920

DT Journal LA Unavailable Page 344

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IT 2622-89-1, Borinic acid, diphenyl-
(detn. of)
RN 2622-89-1 CAPLUS
CN Borinic acid, diphenyl- (6CI, 7CI, 8CI, 9CI) (CA INDEX NAME)
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Ph |
|
Ph— B— OH
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AB In neutral or OAc- soln., Hg++ reacts with B(C6H5)4-, (C6H5)3B, or (C6H5)BOH to form C6H5B(OH)2 and (C6H5)2Hg. The latter reacts with more Hg++ to form C6H5Hg+. Potentiometrically, 2 breaks are found; but amperometrically, only the final end point is detected. Hg++ can be titrated with B(C6H5)4- or C6H5B(OH)2.

ANSWER 274 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN L4AN 1960:68012 CAPLUS 54:68012 DN OREF 54:13036h-i TIDiphenylboric acid from sodium tetraphenylborate ΑU Neu, Richard CS Willmar Schwabe, Karlsruhe, Germany Arch. Pharm. (1959), 292, 437-42 SO DTJournal LΑ Unavailable ΙT 2622-89-1, Borinic acid, diphenyl-

RN

CN

AB cf. CA 49, 9538f. Ph4BNa (I) (300 mg. in 100 ml. H2O) was converted to Ph4BH by passing through a column of acid Lewatit-KSN or Merch I ion exchange resin. Addn. of 8-hydroxyquinoline (300 mg. in 20 ml. MeOH) to the effluent gave 270 mg. Ph2BOH (II), m. 203-6.degree.. The aminoethyl ester of II was prepd. by shaking 3 g. I with the ion exchanger 8 hrs. at room temp., extg. the slurry with EtOH, evapg. the ext. in vacuo and adding 0.5 ml. NH2C2H4OH to yield 1.3 g. ester, m. 184-7.degree..

Borinic acid, diphenyl- (6CI, 7CI, 8CI, 9CI) (CA INDEX NAME)

L4 ANSWER 275 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN AN 1960:68011 CAPLUS

DN 54:68011

OREF 54:13036b-h

TI Study of imines. III. Phosphineiminium chlorides and triphenylphosphine imine

AU Appel, Rolf; Hauss, Alfred

CS Univ. Heidelberg, Germany

(prepn. of)

2622-89-1 CAPLUS

SO Chemische Berichte (1960), 93, 405-11 CODEN: CHBEAM; ISSN: 0009-2940

DT Journal

LA Unavailable

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Ph
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Ph— B— OH
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AB cf. C.A. 53, 9112d. Tertiary phosphines with H2NCl (I) yielded the corresponding phosphineiminium chlorides, [R3PNH2]Cl. [Ph3PNH2]Cl (II) treated with NaNH2 in liquid NH3 yields Ph3P:NH (III), comparable to the isosteric Ph3P:CH2. The reaction of III with polarizable O derivs. results in an exchange of the O by :NH, yielding the corresponding imine derivs. and Ph3PO (IV). A stream of gaseous NH3-I mixt. (250-300 cc.) is condensed at -78.degree. onto 10 g. Ph3P in 100 cc. dry Et2O, the NH3 evapd., and the Et2O removed to give 18 g. mixt. of II and NH4Cl, which extd. with 9:1 EtOAc-MeOH left the insol. NH4Cl and yielded 10.5 q. III, m. 236.degree. (pptd. from EtOAc with petr. ether). Et20 (100 cc.) contq. 136 mg. I treated dropwise with 700 mg. Ph3P in 200 cc. Et2O and the ppt. (812 mg.) dissolved in a little MeOH and repptd. with Et2O yielded 715 mg. II, m. 236.degree.. The aq., neutral soln. of II is stable for some time but deposits IV, needles, m. 156.degree., when treated with a little alkali or acid. MePh2P (10 g.) and I gave in the usual manner 11.8 g. [MePh2PNH2]Cl, m. 167.degree., which, hydrolyzed in the presence of alkali or acid, gave MePh2PO. In the same manner was obtained from 10 g. Me2PhP 12.5 g. [Me2PhPNH2]Cl, m. 106.degree., which was hydrolyzed by acid or alkali to Me2PhPO, m. 100.degree.. Me3P (4 g.) in 50 cc. Et2O treated in the usual manner with I yielded 1.6 g. [Me3PNH2]Cl, m. 122.degree.. Na (722 mg.) and a few crystals of Fe(NO3)3 added under N to about 200 cc. liquid NH3, the mixt. stirred several hrs. until the blue color disappeared, treated with 12 g. powd. II, the NH3 evapd., the residue dissolved in 300 cc. dry C6H6 and filtered from excess II and NaCl, and the filtrate concd. and filtered yielded 7.2 g. III, m. 128.degree. (cyclohexane). The aq. soln. of III reacts strongly basic and becomes turbid after a few sec. with the pptn. of IV. The III was converted with HCl to II and with BzCl to Ph3P:NBz, m. 194.degree.. III (3.55 g.) in 100 cc. dry C6H6 treated 15 min. with a stream of CO2 gave 1.55 g. [Ph3PNH2]NCO, m. 120.degree., hydrolyzed by acid or alkali to IV and NH3. III (2.7 g.) in 100 cc. C6H6 treated dropwise with a slight excess of CS2 gave 1.63 g. [Ph3PNH2]SCN, m. 173.degree.; the C6H6 filtrate worked up yielded 1.42 g. Ph3PS, m. 159.degree.. III (5.85 g.) and 3.4 g. Ph2CO in 200 cc. C6H6 refluxed 2 hrs. and distd. yielded 3.24 g. Ph2C:NH, b1 about 108.degree., which dissolved in C6H6 and treated with dry HCl gave [Ph2CNH2]Cl, sublimes at 230-50.degree.. Ph2C:NH with 2,4-(O2N)2C6H3NHNH2 gave $2,4-(O2N)\ 2C6HNHN:CPh2$, m. 202.degree. III $(2.58\ g.)$ in $100\ cc$. C6H6 treated with $0.86\ g.$ p-MeC6H4SO2Cl in $50\ cc$. C6H6 and filtered, and the filtrate concd. gave 1.9 g. p-MeC6H4SO2N: PPh3, m. 193.degree. (C6H6).

L4 ANSWER 276 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

AN 1960:56332 CAPLUS

DN 54:56332

OREF 54:10967c-e

TI Organoboron compounds. XXXVII. Lithium salts of diarylboronic acids and their complex compounds with dioxane

AU Mikhailov, B. M.; Vaver, V. A.

CS Inst. Org. Chem., Acad. Sci. U.S.S.R., Moscow
SO Zhurnal Obshchei Khimii (1959), 29, 2248-53
CODEN: ZOKHA4; ISSN: 0044-460X
DT Journal
LA Unavailable
LT 62981-91-3 Borinic acid. di-1-paphthyl- 66117-64-4

RN 62981-91-3 CAPLUS

CN Borinic acid, di-1-naphthalenyl- (9CI) (CA INDEX NAME)

RN 66117-64-4 CAPLUS CN Borinic acid, bis(4-methylphenyl)- (9CI) (CA INDEX NAME)

AΒ Diarylboronic acids react like protonic acids in nonag. media. All the reactions below were run under N. Addn. of 0.018 mole BuLi soln. to 5 g. (1-C10H7)2BOH in dry C6H6 gave in 10 hrs. a cryst. ppt. of its Li salt. Similarly, (p-MeC6H4)2BOH (I) and p-MeC6H4Li gave the Li salt of the former acid, a cryst. solid. (o-MeC6H4)2BOH with BuLi in hexane-C6H6 gave a ppt. of mixed (o-MeC6H4)2BBuOH.Li and its cleavage products (o-MeC6H4)2BLi and (o-MeC6H4)BBuOLi. This mixt. treated with dioxane in Et20 gave on evapn. of the org. layer and treatment with isopentane in the cold a ppt. of (o-MeC5H4)2BOLi.O(CH2CH2)2O, which also formed from (o-MeC6H4)2BOH and o-MeC6H4Li.O(CH2CH2)2O. The latter procedure also gave from p-MeC6H4Li.O(CH2CH2)2O and (p-MeC6H4)2BOH in Et2O-dioxane a ppt. of relatively insol. (p-MeC6H4)2BOLi.O(CH2CH2)2O, while the filtrate gave (p-MeC6H4)3BOH.Li.O(CH2CH2)2O. (1-C10H7)2BOH in Et2O formed a dioxane adduct, m. 130-1.degree., which with BuLi gave (1-C10H7)2BOLi.O(CH2CH2)2O. Treatment of (1-C10H7)2BOLi with Me2SO4 in C6H6 gave 72.3% (1-C10H7) 2 BOMe, m. 101-3.degree.. (1-C10H7)2BOLi and MeOH in Et2O gave in 15 min. (1-C10H7)2B(OMe)OH.Li.Et2O.

L4 ANSWER 277 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

AN 1960:56331 CAPLUS

DN 54:56331

OREF 54:10966i,10967a-c

TI Organoboron compounds. XXXVI. Unsymmetric diarylboronic acids and their

Page 347

derivatives

AU Mikhailov, B. M.; Fedotov, N. S.

CS Inst. Org. Chem., Acad. Sci. U.S.S.R., Moscow

SO Zhurnal Obshchei Khimii (1959), 29, 2244-8

CODEN: ZOKHA4; ISSN: 0044-460X

DT Journal

LA Unavailable

IT 109845-79-6, Borinic acid, phenyl-p-tolyl-

(and derivs.)

RN 109845-79-6 CAPLUS

CN Borinic acid, phenyl-p-tolyl- (6CI) (CA INDEX NAME)

IT 62981-91-3, Borinic acid, di-1-naphthyl- 66117-64-4, Borinic acid, di-p-tolyl-

(derivs.)

RN 62981-91-3 CAPLUS

CN Borinic acid, di-1-naphthalenyl- (9CI) (CA INDEX NAME)

RN 66117-64-4 CAPLUS

CN Borinic acid, bis(4-methylphenyl) - (9CI) (CA INDEX NAME)

Cf. C.A. 52, 17148f; 54, 261e, 1266f, 1267a, 8684b. All reactions below were run under N. Reaction of 0.4 mole 1-C10H7MgBr and 88.3 g. PhB(OCH2CHMe2)2 in Et2O at -60.degree. gave after treatment with dil. HCl 80% 1-C10H7BPhOCH2CHMe2 (Ia), b8 207-8.degree., d20 1.099, nD20 1.597; passage of NH3 into an Et2O soln. of this gave the NH3 adduct, m. 90-2.degree. (in sealed tube). Similarly was prepd. 41% (p-MeC6H4)BPhOCH2CHMe2 (I), b1.5 126-8.degree., 0.9720, 1.55.52; NH3

adduct, m. 87-9.degree.. Treatment of I with 100% excess H2NCH2CH2OH gave 87.3% (p-MeC6H4)BPhOCH2CH2NH2, m. 163-5.degree.. PhB(OCH2CHMe2)2 and p-BrC6H4MgBr gave 33% (p-BrC6H4)BPhOCH2CHMe2, b2 152-3.degree., 1.9199, 1.5773. Treatment of 13 g. Ia with 9.4 g. PCl5 with heating to 50-60.degree. gave 76.3% 1-C10H7BPhCl (II), b4 180-1.degree., m. 87-90.degree.. Similarly was prepd. 50% p-MeC6H4BPhCl, b8 142-4.degree., d20 1.5783. Treating I in Et2O with N NaOH with ice cooling gave after evapn. and treatment with isopentane at -40.degree. a low yield of 1-C10H7BPhOH, m. 57-9.degree.. Similarly was prepd. p-MeC6H4BPhOH, an oil, which on standing transformed into 88% (PhBO)3, m. 212-14.degree.. p-MeC6H4BPhCl formed an equimolar adduct with dioxane, m. 72-6.degree.. Similar adduct formed with Ph2BCl, m. 80-5.degree., and with (1-C10H7)2BCl, m. 93-6.degree., and with 1-C10H7BPhCl, m. 90-1.degree.. These are not stable in storage.

L4 ANSWER 278 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

AN 1960:56330 CAPLUS

DN 54:56330

OREF 54:10966e-i

TI Syntheses of .gamma.-lactones and similar compounds in the 1,1-dimethyl-.DELTA.9-octahydronaphthalene series

AU Mousseron-Ganet, M.; Mousseron, M.

CS Ecole natl. superieure chim., Montpelier, Fr.

SO Compt. rend. congr. intern. chim. ind., 31e, Liege 1958 (1959), 2, 625-30; Pub. as Ind. chim. belge, Suppl.

DT Journal

LA French

RN 13331-25-4 CAPLUS

CN Borinic acid, 1-naphthalenylphenyl- (9CI) (CA INDEX NAME)

AB Treatment of 20 g. 4-(2-methyl-2-penten-5-yl)cis-4-cyclohexene-1,2dicarboxylic acid (I) with 60 cc. 98% HCO2H (II) and 4 cc. H2SO4 1 hr. at 80 degree followed by neutralization of H2SO4, distn. of II, extn. of the residue with Et2O, and evapn. of the solvent produced the 7,9-lactone of 1,1-dimethyl-6-carboxy-trans-9,10-decahydronaphthalene-7,9-dicarboxylic acid (III), m. 206.degree. The infrared spectra of III showed the presence of the following bands: .gamma.-lactone, 5.62 .mu.; carboxylic acid, 5.82 .mu.; and the gem-dimethyl group, 7.18 .mu., 7.29 .mu.. Tricyclic oxides having a strong ambergris-like odor were prepd. from the condensation products of myrcene (IV) or 1-methylmyrcene (V) with maleic anhydride (VI) as follows. The reaction of 40 g. VI with 70 g. IV in 200 cc. C6H6 at 50.degree. for 10 hrs. produced the anhydride (VII) of I, m. 34-5.degree., which was then hydrolyzed to I, m. 122-3.degree.. of I with CH2N2 at 0.degree. gave the di-Me ester of I b0.3 138-40.degree., which was then reduced with LiAlH4 to the 1,2-dimethylol analog (VIII) of I, b0.4 160-5.degree.. Distn. of VIII over alumina gave

4-(2-methyl-2-penten-5-yl)-4-cyclohexeno[c]-cis-tetrahydrofuran, b0.5 110-12.degree., which formed 1,1-dimethyl-6,7-tetrahydrofurano-.DELTA.9-octahydronaphthalene (IX), b0.7 100-2.degree., when heated 1 hr. at 80.degree. with II. The reaction of VII with II at 80.degree. produced the anhydride of 1,1-dimethyl-6,7-dicarboxy-.DELTA.9-octahydronaphthalene, m. 98-100.degree., which was hydrolyzed to the corresponding di-acid (X), m. 170.degree.; subsequent treatment of X with II gave the 7-Me deriv. (XI) of IX, b0.5 98-9.degree.. Similar treatment of a series of compds. derived from the reaction of V and VI resulted in the 8-Me deriv. of IX, b0.5 100-5.degree., and the 7,8-di-Me deriv. of IX, b0.1 95-100.degree..

L4 ANSWER 279 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

AN 1960:54027 CAPLUS

DN 54:54027

OREF 54:10512d-e

TI Spectroscopic investigations of condensed phosphates and phosphoric acids. VII. Cubic pyrophosphates

AU Steger, E.; Leukroth, G.

CS Tech. Hochschule, Dresden, Germany

SO Z. anorg. u. allgem. Chem. (1960), 303, 169-76

DT Journal

LA Unavailable

RN 62981-91-3 CAPLUS

CN Borinic acid, di-1-naphthalenyl- (9CI) (CA INDEX NAME)

IT 66117-64-4, Borinic acid, di-p-tolyl- 128711-63-7, Borinic acid, di-1-naphthyl-, compd. with p-dioxane (spectra of)

RN 66117-64-4 CAPLUS

CN Borinic acid, bis(4-methylphenyl) - (9CI) (CA INDEX NAME)

RN 128711-63-7 CAPLUS

CN Borinic acid, di-1-naphthyl-, compd. with p-dioxane (6CI) (CA INDEX NAME)

PAGE 1-A

PAGE 2-A

AB cf. C.A. 54, 2938a. The infrared spectra of SiP2O7, TiP2O7, SnP2O7, and ZrP2O7, and the Raman spectrum of ZrP2O7 are reported. From a consideration of the observed frequency shifts and the limited utility of selection rules of the isolated ion these compds. may well be regarded as double oxides.

L4 ANSWER 280 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

AN 1960:44693 CAPLUS

DN 54:44693

OREF 54:8842f-i,8843a-c

TI Synthesis and structure of aromatic boron compounds

AU Davidson, J. M.; French, C. M.

CS Queen Mary Coll., London

SO Journal of the Chemical Society, Abstracts (1960) 191-5 CODEN: JCSAAZ; ISSN: 0590-9791

DT Journal

LA Unavailable

RN 131732-34-8 CAPLUS CN Borinic acid, 2-biphenylylphenyl- (6CI) (CA INDEX NAME)

AΒ 10-Hydroxy-9-oxa-10-boraanthracene (I) was prepd. and its aromatic character demonstrated by ultraviolet spectroscopy. The mechanism of the reaction of Bu metaborate (II) with Grignard and Li reagents was investigated and the conditions under which org. boronous or boronic acid was the predominant product were examd. An attempt to prep. 9-diethylamino-9-borafluorene was also described. PhMgBr (from 15.7 q. PhBr) in 50 ml. Et20 treated dropwise under reflux with 10 g. phenylboronic anhydride (III) in 75 ml. C6H6, refluxed a further 0.5 hr., the mixt. hydrolyzed with 200 ml. 15% HCl, the solvents removed, 20 ml. ligroine added, and the mixt. filtered gave 3.5 g. III, m. 214.degree.. Removal of the solvent from the filtrate gave 11.5 g. diphenylboronous acid (IV), n20D 1.5907. IV with HOCH2CH2NH2 formed 65% 2-aminoethyl diphenylboronite, m. 187.degree.. 2-Biphenylylphenylboronous acid (V) was similarly prepd. front 7.5 g. III and 1 mole 2-biphenylylmagnesium iodide in Et2O, after hydrolysis, ethanolamine added, and crystd. to give 10.2 g. 2-aminoethyl-2-biphenylyl phenylboronite (VI), m. 175.degree. (alc.). VI (3 g.) shaken with 30 ml. Et20 and 30 ml. 10% HCl gave 2.55 g. V, viscous liquid. Mg (0.7 g.) reacted readily with 7.5 g. 2-iododiphenyl ether and 3 g. II in 60 ml. Et20 after addn. of iodine; after 10 min. of spontaneous refluxing and 0.5 hr. of heating the mixt. was hydrolyzed with 100 ml. 15% HCl, the acid products extd. with 5% NaOH, and the basic ext. acidified to give 1.5 g. o-phenoxyphenylboronic acid, m. 114.degree. (C6H6-cyclohexane). 9-Bromophenanthrene (5 g.) and 2.5 g. II gave 2.45 g. 9-phenanthrylboronic acid, m. 324.degree. (H2O). 2,2'-Dilithiodiphenyl ether in 156 ml. Et20 treated during 10 min. with 6.7 g. II in 25 ml. Et20, the soln. refluxed 2 hrs., and hydrolyzed with 100 ml. 10% HCl gave 5.9 g. 10-hydroxy-9-oxa-10-boraanthracene (VII), m. 285.degree. (C6H6-cyclohexane). The same soln. of 2,2'-dilithiodiphenyl ether (600 ml.) and 200 ml. ether soln. contg. 37 g. BF3-Et20 simultaneously added to 100 ml. Et20 under N during 45 min. and the mixt. refluxed 1 hr. gave 11.1 g. VII. 2-Biphenylylmagnesium iodide (from 10 g. 2-iodobiphenyl) in 50 ml. Et20 treated rapidly with 3.5 g. II in 15 ml. Et20, and the soln. refluxed 0.5 hr. gave 5 g. 2-biphenylylboronic acid (VIII), m. 121-3.degree. (H2O), resolidified to the anhydride, m. 195.degree.. VIII

(3.9 g.) esterified with alc. by azeotropic distn. gave 3.4 g. di-Et ester, b4 136-8.degree., n20D 1.5444. Di-Bu 2-biphenylylboronate (IX) was prepd. by direct esterification of the Grignard reaction mixt. after hydrolysis. 2-Iodobiphenyl (26.5 g.) and 8.8 g. II afforded 13.3 g. IX, b0.6 149-51.degree., n20D 1.5310. IX (7.5 g.) heated 18 hrs. at 140.degree. with 11 g. PCl5 gave 4.35 g. 2-biphenylylboron dichloride, b0.25 95-6.degree., n20D 1.5661. 2,2'-Dilithiobiphenyl (from 4 g. 2,2'-diiodobiphenyl) in 60 ml. Et2O slowly treated with 0.9 g. II in 15 ml. Et2O under N, the soln. refluxed 15 min., hydrolyzed with dil. NH4Cl, and the soln. azeotropically distd. with HOCH2CH2NH2 and PhMe gave 1.3 g. bis(2-aminoethyl)2-biphenylyl boronate, m. 134.degree. (C6H6). A sample was hydrolyzed with dil. HCl to the acid which was dried to form the anhydride, m. 206.degree. (cyclohexane).

L4 ANSWER 281 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

AN 1960:40329 CAPLUS

DN 54:40329

OREF 54:7964h-i,7965a

TI Compositions for controlling plant growth

IN Barnsley, Geoffrey E.; Eaton, John K.; Airs, Raymond S.

PA "Shell" Research Ltd.

DT Patent

LA Unavailable

FAN.CNT 1

ΡI

PATENT NO. KIND DATE APPLICATION NO. DATE

GB 818925 19590826 GB

RN 2622-89-1 CAPLUS

CN Borinic acid, diphenyl- (6CI, 7CI, 8CI, 9CI) (CA INDEX NAME)

RN 89566-59-6 CAPLUS
CN Borinic acid. bis(4-chlore

CN Borinic acid, bis(4-chlorophenyl)- (9CI) (CA INDEX NAME)

AB Organoboron acids and their anhydrides, esters, and complexes with nitrogenous bases are effective as pre- or postemergence weed killers. Suitable compds. are halo-, methyl-, halomethyl-, and methoxy-substituted phenylboronic and diphenylborinic acids, and unsubstituted diphenylborinic acid. Cl is the preferred halogen substituent. More than 1 substituent per phenyl group may be used if 1 position ortho to the B atom is unsubstituted. The choice of substituents permits selective toxicity.

Thus, bis(4-chlorophenyl)borinic acid is highly toxic to both mono- and dicotyledons, whereas diphenylborinic acid is toxic to dicotyledons and inert to monocotyledons. The compds. are applied as mixts. with a surface-active agent, an inert solid carrier, or an inert liquid carrier which can be a solvent for the organoboron compd.

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ANSWER 282 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN
L4
AN
     1960:34015 CAPLUS
DN
     54:34015
OREF 54:6598d-i
TI
     Preparation, properties and uses of benzeneboronic acid
ΑU
     Washburn, Robert M.; Levens, Ernest; Albright, Charles F.; Billig,
     Franklin A.; Cernak, E. S.
CS
     Am. Potash & Chem. Corp., Whittier, CA
    Advances in Chem. Ser. (1959), 23, 102-28
SO
DT
     Journal
LΑ
     Unavailable
ΙT
     2622-89-1, Borinic acid, diphenyl-
        (formation in reaction of Me borate with PhMgBr)
RN
     2622-89-1 CAPLUS
     Borinic acid, diphenyl- (6CI, 7CI, 8CI, 9CI) (CA INDEX NAME)
CN
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Ph | Ph— B— OH

The prepn. of PhB(OH)2 (I) from PhMgBr (II) and (MeO)3B (III) was AΒ investigated as to reaction temp., rate of addn. of II to III, concn. of reactants, effect of impurities in III, and method of isolation of I. When all other variables were kept const., the yield of I was improved by adding 3.0 moles II in 0.33 hr. as compared to 4.0 hrs., increased from about 53% to about 64% by decreasing the mole-% III from about 17 to 4, increased by lower temps. (52.4% at 0.degree., 61.2% at -30.degree., and 85.3% at -60.degree.), and unaffected when II was added to III or III added to II. These investigations led to the following incremental procedure for the prepn. of I. III (336 q.) was distd. directly into a buret; 1500 ml. anhyd. Et20 was placed in the reaction flask, cooled to O.degree., 1000 ml. 3N II placed in a 2nd buret, and with vigorous agitation and under N, while maintaining the temp. at 0.degree., 10 ml. portions of III and 30 ml. portions of the II added as increments. The addn. of both reagents was complete in 0.25 hr., the whole stirred 0.33 hr., 200 ml. H2O added in 5 min. at 0.degree., the mixt. neutralized with 84 ml. H2SO4 in 1700 ml. H2O at 0.degree., the Et2O layer sepd., the H2O layer extd. with 3 250-ml. portions of Et20, and the combined Et20 solns. transferred to a 5-1. flask equipped with Hershberg stirrer, modified Claisen still-head, and dropping funnel. Part of Et20 was distd., 1500 ml. H2O added slowly, and distn. continued until the still head temp. reached 100.degree.. While stirring, the distilland was cooled rapidly to 11.degree. and filtered to give 277 g. I. Only traces of Ph2BOH were obtained. Using these conditions p-ClC6H4MgBr gave 81% p-ClC6H4B(OH)2 and 1-C10H7MgBr gave 68% 1-C10H7B(OH)2. I was readily converted to the anhydride (IV); spectroscopically pure I was obtained by crystn. from H2O followed by drying either by a stream of air almost satd. with H2O or by keeping overnight in open dish at ordinary temps. in 30-40% relative humidity. I could not be recrystd. from C6H6 without partial conversion to IV; the m.p. could not be used as a criterion of purity. I in PhMe

under azeotropic distn. conditions gave IV. Infrared spectra of pure I and IV were given; the bands at 1023, 1086, and 1175 cm.-1 were characteristic of IV. The soly. of I in H2O, MeOH, Et2O, C6H6, xylene, CCl4, and petr. ether and IV in C6H6, PhMe and xylene was shown graphically. The literature on the reaction of I was reviewed. 89 references.

L4 ANSWER 283 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN .

AN 1960:7244 CAPLUS

DN 54:7244

OREF 54:1519e

TI Direct preparation of diphenylboric acid complexes

AU Neu, Richard

SO Naturwissenschaften (1959), 46, 262-3

CODEN: NATWAY; ISSN: 0028-1042

DT Journal

LA Unavailable

IT 2622-89-1, Borinic acid, diphenyl-

(amine complexes)

RN 2622-89-1 CAPLUS

CN Borinic acid, diphenyl- (6CI, 7CI, 8CI, 9CI) (CA INDEX NAME)

AB Various amines such as 8-hydroxyquinoline, 1-aminoethanol, and pyridine-2-carbinol react with sodium tetraphenylborate on heating in EtOH, acetone, or water to give the diphenylboric acid complex. The reaction is suggested as a possible differential test for amines.

L4 ANSWER 284 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

AN 1959:118507 CAPLUS

DN 53:118507

OREF 53:21157i,21158a

TI Amino alcohol esters of hydroxy boranes. II. Infrared spectra of boroxazolidines, and nitrogen-boron coordination

AU Weidmann, Hans; Zimmerman, Howard K., Jr.

SO Ann. (1959), 620, 4-7

DT Journal

LA Unavailable

IT 2622-89-1, Borinic acid, diphenyl-

(aminoalkyl esters (cyclic form), spectra of)

RN 2622-89-1 CAPLUS

CN Borinic acid, diphenyl- (6CI, 7CI, 8CI, 9CI) (CA INDEX NAME)

AB cf. C.A. 53, 17144d. Infrared spectra are schematically shown for 12 compds. of amino alcs. with hydroxy boranes, covering the range, 700-1700 cm.-1 The behavior of an absorption band at about 1200 cm.-1 is attributed to the presence of N-B coordination in these compds. in the

solid state.

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ANSWER 285 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN
L4
AN
     1959:94837 CAPLUS
DN
     53:94837
OREF 53:17144d-i,17145a
     Boric acid esters of N-substituted amino alcohols
TI
AU
    Weidmann, Hans; Zimmerman, Howard K., Jr.
CS
    A. & M. Coll. of Texas, Texas Station
    Ann. (1958), 619, 28-35
SO
DT
     Journal
LΑ
    Unavailable
IT
     2622-89-1, Borinic acid, diphenyl-
        (esters (cyclic form))
RN
     2622-89-1 CAPLUS
CN
     Borinic acid, diphenyl- (6CI, 7CI, 8CI, 9CI) (CA INDEX NAME)
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Ph | | | Ph— B— OH

GI For diagram(s), see printed CA Issue.

AΒ H3BO3 and mono- and disubstituted boric acids could be esterified just as readily with N-substituted as with unsubstituted amino alcs. In contrast to the usual trialkyl or triaryl borates which are very sensitive to H2O and undergo hydrolysis readily, the borates of N-substituted amino alcs. show greater stability which is ascribed to a N .fwdarw. B linkage with the formations of boroxazolidines (I, II, and III) or the tetrahydroboroxazine (IV). Quant. data are given for solubilities of I, II, III, and IV in dioxane, C6H6, Et2O, AcOEt, Me2CO, MeCN, H2O, and HCONH2; in most instances soly. in MeOH and EtOH was also detd. studies in aq. media indicate that a zwitterion is formed when the N .fwdarw. B bond undergoes hydrolysis. Despite their resistance to hydrolysis, I, II, III, and IV when treated with picric acid in Et2O or CHCl3 yielded, in all cases, the picrates of the corresponding amino alcs. The mechanism of this degradation was not explained. Usually I were formed by mixing the appropriate amino alc. (V) in aq. EtOH with Ph2BOH (VI) in Et20; in 1 instance, equimolar amts. of V and VI were heated at 90.degree. in vacuo, dissolved in CH2Cl2, treated with C, filtered, evapd. and crystd. from C6H6. The following I, in which R1 and R2 = Ph, were prepd. (V, % yield, R3, R4, and R5, and m.p. of I given): HOCH2CH2NH2, 67, H,H,H, 190-2.degree.; HOCH2CH2NHMe, 50, Me,H,H, 193-5.degree.; HOCH2CH2NMe2, 84, Me, Me, H 166-7.degree.; HOCH2CH2NHEt, 67, Et, H, H, 188-90.degree.; H2NCH2CHMeOH, 63, H,H,Me. From R1B(OH)2 (R1 = Ph or Bu) the following II were formed (V, R1 and R3, % yield, and m.p. given): (HOCH2CH2)2NH, Ph, H, 70, 209.5-10.degree.; (HOCH2CH2)2NMe, Ph, Me, 100, 104-7.degree.; (HOCH2CH2)2NEt, Ph, Et, 100, 96-99.degree.; (HOCH2CH2)2NH, Bu, H, 61, 153-5.degree.. N(CH2CH2OH)2 and H3BO3 were esterified under reduced pressure and treated with CH2Cl2 to give 58% IV (R1 = R2 = Ph, R3 = R4 = H), m. 190-2.degree. (50% EtOH or C6H6). The soly. of various I and II in 9:1 H2O-MeCN was a function of the pH. The min. soly. was attained at about pH 2-5; the max. at about pH 6. Potentiometric titrations of I (R1 = R2 = Ph, R3 = R4 = R5 = H) gave rise to a ppt. at about pH 6.5, which redissolved above or below this pH.

L4 ANSWER 286 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

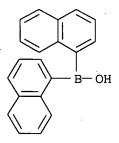
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Page 356

AN 1959:72434 CAPLUS DN 53:72434 OREF 53:13107d-i ΤI Organoboron compounds Letsinger, Robert L.; Skoog, Ivan H.; Remes, Nathaniel L. ΙN PA Callery Chemical Co. DTPatent LΑ Unavailable FAN.CNT 1 PATENT NO. KIND DATE APPLICATION NO. DATE _____ _____ US 2872479 PΙ 19590203 US IT 2622-89-1, Borinic acid, diphenyl-(and derivs.) RN 2622-89-1 CAPLUS Borinic acid, diphenyl- (6CI, 7CI, 8CI, 9CI) (CA INDEX NAME) CN

Ph | Ph— B— OH

RN 62981-91-3 CAPLUS CN Borinic acid, di-1-naphthalenyl- (9CI) (CA INDEX NAME)



AB Dialkyl and diarylhydroxyboranes which are useful as oil additives, antioxidants, and synthesis intermediates can be prepd. in a pure state by sepg. them from mixts. as an ethanolamine or ethylene glycol deriv. and then hydrolyzing the deriv. to yield the pure borane. Thus, PhMgBr in

Et2O (1.2M, 475 cc.) was added slowly under N to triisobutoxyborane (0.284M) in 150 cc. Et2O. After refluxing 2 addnl. hrs. the soln. was hydrolyzed with 200 cc. 3.6M HCl. The Et2O layer was washed and dried with CaSO4 and then distd. Up to 150.degree./15 mm., biphenyl (0.62 g.) was recovered, m. 67-8.degree.. The remaining liquid distd. at 150-205.degree./15 mm. and triphenylborane remained as a solid. After the liquid was dild. with Et2O, it was satd. with NH3 to ppt. ammoniadiphenylisobutoxyborane, m. 64-7.degree. (I). I (2.13 g.) was heated with 2 cc. ethanolamine in 100 cc. PhMe, distd. until only 10 cc. remained and the residue cooled to give 1.67 g. diphenyl (2aminoethoxy)borane (II), m. 187-8.degree.. Also, 13.7 g. I in 100 cc. EtOH and 200 cc. H2O mixed with 6 cc. ethanolamine gave 10.91 g. II. (5.33 g.) in MeOH and Me2CO was acidified with HCl, H2O added and the diphenylhydroxyborane extd. with Et2O, dried with MgSO4, and vacuum distd. to yield 2.11 g. bis(diphenylboryl) oxide, b1 210-13.degree., m. 104-5.degree.. Or 948 cc. PhMqBr (1.94 M in Et20) was added to 113 q. 4,5-dihydro-2-butoxy-2-bora-1,3-dioxole (III), the mixt. kept overnight, then hydrolyzed with 600 cc. 3.4M HCl, the Et2O layer dried and split into 2 parts, 1 of which was added to 20 cc. BuOH in 800 cc. PhMe. On distn. at 124-60.degree./2 mm., diphenylbutoxyborane was isolated and was then treated as above. By similar techniques di(1-naphthyl)hydroxyborane, m. 105-6.degree., and di-1-naphthyl(2-aminoethoxy)borane, m. 205-6.degree., were prepd. 1-Naphthylmagnesium bromide (0.05M) was added to 11.7 q. phenyldibutoxyborane in 120 ml. Et2O, the mixt. kept overnight, then hydrolyzed with dil. HCl, the Et2O layer sepd., and the Et2O evapd. To the residue was added 125 cc. EtOH, 20 cc. H2O, and 6 cc. ethanolamine and the ppt. recrystd. (Me2CO-H2O) to give phenyl-1-naphthyl-(2aminoethoxy) borane, m. 228-9.degree.. On acid hydrolysis, phenyl-1-naphthylhydroxyborane was isolated. On cleavage with ZnCl2, H2O2, or Br2, 1-HOC8H9, C8H10, and PhCO2H were isolated. When 91.6 g. III was added to BuMgBr (0.636M) at -60 degree. over 4 hrs., the mixt. hydrolyzed and the Et20 layer sepd. and added to 0.63M ethylene glycol in 1 l. PhMe, 1,2-bis(dibutylboryloxy)ethane (IV) was isolated after distn. at 133-4.degree.. IV was hydrolyzed with NaOH, Et2O added, the H2O layer acidified, and the Et2O evapd. to give dibutylhydroxyborane; this on adding PhMe and distg., formed bis(dibutylboryl) oxide, b1, 102.degree.. Similarly, isopentylmagnesium bromide and III formed 1,2bis (diisopentylboryloxy) ethane, b0.5 147-8.degree. and diisopentylhydroxyborane.

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L4 ANSWER 287 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN
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AN 1959:23302 CAPLUS

DN 53:23302

OREF 53:4270f-i,4271a-g

TI Amine boranes. II. The preparation of pyridine diarylboranes

AU Hawthorne, M. Frederick

CS Rohm & Haas Co., Redstone Arsenal, Huntsville, AL

SO Journal of the American Chemical Society (1958), 80, 4293-6 CODEN: JACSAT; ISSN: 0002-7863

DT Journal

LA Unavailable

RN 96484-29-6 CAPLUS

CN Borinic acid, bis(4-bromophenyl) - (9CI) (CA INDEX NAME)

RN 73774-45-5 CAPLUS

CN Borinic acid, bis(4-methoxyphenyl) - (9CI) (CA INDEX NAME)

AB The low temp. reduction of Bu, Et, and H2N(CH2)2 diarylborinates with LiAlH4 in Et20 contg. pyridine gave modest yields of pyridine diarylboranes. An attempt to prepare similarly pyridine-1-C10H7PhBH was unsuccessful. Ph2BO(CH2)2NH2 (40.0 g.) in the min. vol. of 1:1 Me2CO-MeOH treated with 20 cc. concd. HCl, dild. with H2O, extd. with Et2O, the ext. washed, dried, evapd., the oily residue dissolved in 100 g. abs. EtOH and 220 g. C6H6, the soln. distd. azeotropically, and the residual oil fractionated gave 26.5 g. Ph2BOEt (I), b. 100.degree.. LiAlH4 (5.00 g.) in 500 cc. dry Et20 refluxed under N, the soln. cooled to -70.degree., dild. with 50 cc. pyridine, treated slowly with stirring at -70.degree. with 25 g. I in 100 cc. dry Et2O, warmed with stirring to 0.degree., dild. with cooling with 10 cc. pyridine and 25 cc. H2O, filtered, the residue washed with Et20, the combined Et20 solns. washed, dried, evapd. at -10.degree., the cryst. residue dissolved in a small amt. of C6H6 at 45.degree., and the soln. dild. with a small vol. of Et20 and then with pentane to turbidity and cooled slowly to 10.degree. yielded 15 g. Ph2BH.C5H5N (II), m. 106-7.degree.. The Grignard reagent from 29 g. p-MeOC6H4Br in 350 cc. dry Et2O and 12 g. Mg added slowly to 58 g. B(OBu)3 (III) in 500 cc. Et2O at -70.degree. under N during 3 hrs., warmed overnight with stirring to room temp., decompd. with a slight excess of HCl, the Et20 layer worked up and distd., the distillate, b0.5 150-200.degree., (36.4 g.) dissolved in 75 cc. EtOH, the soln. treated with 15 cc. H2N(CH2)2OH in 30 cc. H2O, heated 10 min. on the steam bath, cooled, and filtered gave 10.0 g. (p-MeOC6H4)2BO(CH2)2NH2 (IV), m. 168-70.degree.. A small amt. of IV treated with dil. HCl gave (p-MeOC6H4)2BOH, m. 107.degree.. IV (6 g.) in 50 cc. pyridine added slowly to 3.0 g. LiAlH4 in 50 cc. Et2O and 100 cc. pyridine under N at -70.degree., stirred 1 hr. at -20.degree., and worked up in the usual manner yielded 4.1 g. (p-MeO C6H4)2BH.C5H5N, m. 109-10.degree. (C6H6-Et2O-pentane). The Grignard deriv. from 184 g. p-ClC6-H4Br and 24 g. Mg in 400 cc. Et2O added slowly under N to 116 g. III in 1000 cc. dry Et20 with stirring at -70.degree., kept overnight, decompd. with dil. HCl, worked up, the residual oil distd., the distillate (85 g.), b1 150-80.degree., dissolved in 500 cc. BuOH, and the soln. fractionated gave 29 g. p-ClC6H4B(OBu)2, b0.7 118.degree., and 30 g. (p-ClC6H4)2BOBu (V), b0.5 150.degree.. V (6 g.) in 50 cc. dry Et20 treated in the usual manner at -70.degree. with 2.0 q. LiAlH4 and 10 cc. pyridine in 200 cc. Et20 yielded 2.2 g. (p-ClC6H4)2BH. C5H5N, m. 103-4.degree. (C6H6-pentane).

Crude (p-BrC6H4)2BO(CH2)2NH2 (26 g.) treated with excess dil. HCl in the presence of 1000 cc. Et20, the Et20 layer worked up, the oily residue distd. azeotropically with 65 g. abs. EtOH and 150 cc. C6H6, and the residue distd. yielded 10.5 g. pure (p-Br C6H4)2BOEt (VI), b0.25 160.degree.. VI (9 g.) reduced in the usual manner with LiAlH4 in the presence of pyridine yielded 4.2 g. (crude) (p-Br C6H4)2BH.C5H5N, m. 122-4.degree. (C6H6-pentane). The Grignard reagent from 171 g. p-MeC6H4Br and 23 g. Mg in 500 cc. Et2O added under N at -70.degree. slowly to 115 g. III in 500 cc. Et20 with stirring, kept overnight, and worked up in the usual manner yielded 15.3 g. p-MeC6H4B(OBu)2, b0.6 110.degree., and 35 g. (p-Me C6H4)2BOBu (VII), b0.6 138.degree.. VII (6 g.) reduced with LiAlH4 in the presence of pyridine gave 2.6 g. (crude) (p-Me C6H4)2BH.C5H5N, m. 110-13.degree. (C6H6-pentane). Ph(1-C10H7)BO(CH2)2NH2 (8.0 g.) and 50 cc. dry pyridine treated at -20.degree. with 2.0 g. LiAlH4 in 100 cc. dry Et20, stirred 1 hr. at -20.degree., warmed to 0.degree. dild. with 10 cc. H2O in 20 cc. pyridine, faltered, the filtrate washed, dried, evapd., and the oily residue (about 4.0 g.) dissolved immediately with gassing in a small amt. of warm C6H6 and dry Et2O and cooled to -70.degree. deposited a cryst. solid which showed both B-H and strong O-H stretch in the infrared; the material melted over an indefinite range and smelled strongly of pyridine; the enhanced instability of this borane-pyridine is possibly the result of steric crowding of the 3 large groups attached to the tetrahedral B atom. II (2.45 g.) in 10 cc. MeCN treated with 5.0 g. AgClO4 in 2 cc. H2O and 10 cc. MeCN, kept 2 hrs. at room temp., and filtered yielded 0.65 g. Ag. II (4.91 g.) in 60 cc. MeCN and 5 cc. H2O refluxed gave 95% H; the soln. dild. with 300 cc. H2O, extd. with Et2O, and the ext. worked up yielded 4.1 g. Ph2BOH. II (245 mg.) in 10 cc. pyridine in 4:1 pyridine-H2O titrated with 0.020M iodine in pyridine indicated 98% purity. Pure II (12.2 g.) added to 5.3 g. pure iso-BuCl in 50 cc. dry Et20, refluxed 5 hrs. with stirring, cooled to room temp., filtered, treated with 200 cc. EtOH, 20 cc. H2O, $4.0\ g.$ 2,4-(O2N)2C6H3NHNH2, and 2 cc. concd. HCl, heated 1 hr. with stirring on the steam bath, dild. with 1 l. H2O, filtered, the residue extd. with CH2Cl2, the ext. concd. to 20 cc. and chromatographed on Al2O3, the 1st bright yellow band eluted with CH2Cl2, and the eluate evapd. gave 6.3 g. 2,4-(O2N)2C6H3NHN;CHCHMe2, m. 181-2.degree. (aq. EtOH).

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L4
    ANSWER 288 OF 309
                        CAPLUS COPYRIGHT 2003 ACS on STN
ΑN
     1959:6704 CAPLUS
     53:6704
OREF 53:1203i,1204a-b
TI
     Preparation of bromides of organoboron compounds from esters of
     organoboron acids and organoboron chlorides
ΑU
    Mikhailov, B. M.; Blokhina, A. N.; Fedotov, N. S.
CS.
    N. D. Zelinskii Inst. Org. Chem., Moscow
SO
     Izvestiya Akademii Nauk SSSR, Seriya Khimicheskaya (1958) 891-3
     CODEN: IASKA6; ISSN: 0002-3353
DT
     Journal
LΑ
     Unavailable
ΙT
     2622-89-1, Borinic acid, diphenyl-
        (esters, reaction with PBr3 and PBr5)
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Borinic acid, diphenyl- (6CI, 7CI, 8CI, 9CI) (CA INDEX NAME)

Ph | | Ph— B— OH

2622-89-1 CAPLUS

Patel

RN

CN

Cf. Michaelis and Becker, Ber. 13, 58(1880). Heating 1.5 hrs. 23.8 g. Ph2BOCH2CHMe2 (I) and 220 g. PBr3 (II) with 43 g. PBr5 (III) at 130-40.degree. until the red color had been destroyed gave 9 g. PhBBrOCH2CHMe2, bl3 115-19.degree., and 6.4 g. Ph2BBr (IV), b8 150-3.degree., along with PhBr and iso-BuBr. Similarly 36.5 g. III and 18 g. Ph2BOEt in 260 g. II gave 7 g. IV. Heating 35 min. 23.8 g. I with 43 g. III at 130-40.degree. gave 52% iso-BuOBPhBr (V), b8 106-10.degree., and 16.4% IV, b8 150-5.degree., along with iso-BuBr, PhBr, II, and POBr3. Similarly 23.4 g. (iso-BuO)2BPh, 240 g. II, and 43 g. III gave 41.6% V, b9 112-12.5.degree., d20 1.243, n20D 1.5190; when II was omitted the yield was 31.4%. Passage of dry HBr into Ph2BCl 1 hr. at room temp. gave 75% IV, b9 153-4.degree., d20 1.302, n20D 1.6325. PhBCl2 and HBr similarly gave in 6 hrs. 34.6% PhBBr2, b7 78-80.degree., m. 31-2.degree., d30 1.698.

L4 ANSWER 289 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

AN 1958:113359 CAPLUS

DN 52:113359

OREF 52:20003a-c

TI Back-coordination from oxygen to boron in organoboron compounds

AU Abel, E. W.; Gerrard, W.; Lappert, M. F.; Shafferman, R.

CS Northern Polytech., London

SO Journal of the Chemical Society, Abstracts (1958) 2895-7 CODEN: JCSAAZ; ISSN: 0590-9791

DT Journal

LA Unavailable

IT 2622-89-1, Borinic acid, diphenyl-(esters, compds. with pyridine)

RN 2622-89-1 CAPLUS

CN Borinic acid, diphenyl- (6CI, 7CI, 8CI, 9CI) (CA INDEX NAME)

AB Complexes were formed between compds. of the form (RO)xBR'y(x + y = 3) and pyridine, NH3, EtNH2, Et2NH, and Et3N. In (RO)3B when R was CCl3CH2 no complexes were formed; however when R was CF3CH2 stable complexes were formed. While PhB(OR)2 and Bu2B(OR) formed stable complexes, BuB(OR)2 did not. A theoretical discussion is given. In (RO)xBr'y(x + y = 3) R and R' represent the following radicals: Me, Et, CCl3CH2, CF3CH2, Pr, Bu, Ph, 2-C6H4Y, and 2,6-C6H3Y2 where Y = Me, MeO, NO2, and I. Tris(2,2,2-trifluoroethyl) borate, b. 43.degree./60 mm. nD20 1.297, and 2,2,2-trifluoroethyl diphenylboronite, b. 90.degree./0.2 mm., nD20 1.5190, d20 1.1706, were formed by allowing BCl3 and Ph2BCl, resp., to react with the corresponding alcohol.

L4 ANSWER 290 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

AN 1958:113351 CAPLUS

DN 52:113351

OREF 52:20000b-h

TI Properties of the anhydride and esters of diphenylboronous acid

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ΑU
     Abel, E. W.; Gerrard, W.; Lappert, M. F.
CS
     Northern Polytech. Inst., London
SO
     Journal of the Chemical Society, Abstracts (1957) 3833-8
     CODEN: JCSAAZ; ISSN: 0590-9791
DT
     Journal
     Unavailable
LΑ
IT
     2622-89-1, Borinic acid, diphenyl-
        (esters)
RN
     2622-89-1 CAPLUS
CN
     Borinic acid, diphenyl- (6CI, 7CI, 8CI, 9CI) (CA INDEX NAME)
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(prepn. of

cf. preceding abstr. The present paper reports continued investigation of Ph2BOH and its derivs. Ph2BY (where Y is a univalent atom or group), and the prepn. and properties of Ph2BCl (cf. C.A. 51, 2444d), and esters, Ph2BOR (cf. C.A. 51, 9513c). The reactions of (Ph2B)2O (hydrolysis with BBr3 and PCl5 or PBr5) and of Ph2BOR [hydrolysis and reactions with H, B, and P halides (Cl, Br)] are reported. Information is given about the steric course of reactions at the 1-C atom of the R chain; certain reactions of Ph2BOR were carried out with optically active compds. (+)-1-methylheptyl ester was used as one generally responsive to SN2 replacement and the (+)-1-phenylethyl ester responsive to SN1 replacement. Ph2BOR were easily hydrolyzed by cold H2O, NaOH, or aq. HCO2H to Ph2BOH. Of the 3 butyl (n-, sec-, and tert-) diphenyl boronites only the tert-Bu ester reacted with HCl to give Ph2-BOH. (+)-Ph2BOCHPhMe gave much racemized ClCHPhMe. HBr had no effect on the (-)-1-methylheptyl ester, but when the reactants were mixed in the liquid state (-80.degree.) reaction took place at 20.degree. under pressure and C6H6 and (Ph2B)20 were isolated instead of Ph2BOH. BCl3 reacted with Ph2BOBu according to Ph2BOR + BCl3 .fwdarw. Ph2BCl + ROBCl2. A similar reaction to obtain Ph2BCl was shown with n-C8H17OBPh2 and BBr3.3ROBBr2 .fwdarw. 3RBr + BBr3 + B2O3. PBr5 also reacted with diphenylboronites to give Ph2BBr; Ph2BOR + PBr5 .fwdarw. Ph2BBr + RBr + POBr3. (Ph2B)20 was hydrolyzed by acid. With B trihalides or PX5, the Ph2BrX (X = Cl, Br) were obtained. With BX3 the reaction was rapid at low temps., but with PX5, the yields were low even at high temps. Esterification of (Ph2B)20 with ROH having a highly electron-releasing group involves B-O and not C-O fission. Hydrolysis of Ph2BOR involves B-O fission since retention of configuration was observed. The reaction of Ph2BOR with HX indicates SN1 and SN2 mechanisms, depending on the nature of the alkyl group. Evidence for SN2 or SN1 mechanisms are discussed. The ease of B-Cl fission in Ph2BCl is of the same order as that of chloroboronates, but lower than in Cl2BOR and BCl3. Addnl. methods for prepn. of Ph2BCl are given: Ph2BOR + BCl3 and (Ph2B)2O + BX3. The spectra of Ph2BOH, (Ph2B)2O, and Ph2BOR were observed in the 650-5000 cm.-1 region. In all Ph2B compds. there is a splitting of out-of-plane C-H vibration at levels of the Ph group by about 20 cm.-1 which suggests coupling of modes. The B-O stretching modes in Ph2BOH and esters is 1325 .+-. 2 cm.-1 In (Ph2B)20 two bands attributable to B-O stretching modes occur at 1262 and 1378 cm.-1, which are the expected in-phase and out-of-phase modes of B-O-B grouping. Prepns. and techniques are given.

L4 ANSWER 291 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

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AN
     1958:113350 CAPLUS
DN
     52:113350
OREF 52:19999f-i,20000a-b
     Preparation and properties of di-n-butylboronous anhydride
ΑU
     Gerrard, W.; Lappert, M. F.; Shafferman, R.
CS
     Northern Polytech. Inst., London
SO
     Journal of the Chemical Society, Abstracts (1957) 3828-33
     CODEN: JCSAAZ; ISSN: 0590-9791
DT
     Journal
LΑ
     Unavailable
IT
     2622-89-1, Borinic acid, diphenyl-
        (esters)
     2622-89-1 CAPLUS
RN
     Borinic acid, diphenyl- (6CI, 7CI, 8CI, 9CI) (CA INDEX NAME)
CN
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Ph Ph-B-OH

(prepn. of

AΒ cf. following abstr. This is the first of a series of papers designed to outline systematically the chemistry of the dialkylboron system, R2BY. This particular paper is concerned mainly with the chemistry of (Bu2B)20 (I) which was obtained in 56% yield by interaction of 2 moles BuMgBr with 1 mole BF3-Et20, followed by acid hydrolysis and distn.: 2BuMgBr + BF3 .fwdarw. Bu2BOH .fwdarw. 1/2I (A). I was directly esterified to the boronite; (Bu2B)2O + 2ROH .fwdarw. 2Bu2BOR + H2O (B), where R was Bu, iso-Bu, sec-Bu, 1-methylheptyl, allyl and Ph; the method was not applicable for the prepn. of the tert-Bu ester. The (+)-1-methylheptyl ester was also prepd. from (+)-2-octanol with Bu2BCl by the general-type reaction: Bu2BCl + ROH .fwdarw. Bu2BOR + HCl (C). The Bu ester was hydrolyzed with excess water by azeotropic distn. whereas the 1-methylheptyl ester required aq. NaOH. I was readily hydrolyzed by cold H2O and at low pressure the reaction proved to be reversible. The Bu2BX were formed readily by adding BX3 (X = Cl or Br) along with the decompn. of the unstable B oxyhalide. Bu2BCl was also obtained from PCl5 and I. This method was found to be the superior of the two, since no decompn. of the intermediates takes place during the distn. of the chloride. Both halides formed 2:1 complexes, (C5H5N)2.Bu2BX, with pyridine. By slow addn. of an ether soln. of H2O they give either the acid, Bu2BOH, or I, depending on the proportions used. Heating I in excess AcOH produced the novel [Bu(AcO)B]20, characterized by hydrolysis with cold H2O to BuB(OH)2. I did not react with pyridine, Et2NH, NH3, SOCl2, or HCl. Reaction (C) must have taken place without 1 R-O fission; it follows that esterification (B) did not involve R-O fission. Since hydrolysis of (+)-1-methylheptyl dibutylboronite gave (+)-2-octanol having the same rotation as the alc. used, this reaction must have involved B-O fission. The at. refractivity for B, in esters, calcd. by using Vogel's at. refractivities, was 1.95 .+-. 0.15, compared with 4.39 in diphenyl esters. The formation of [Bu(AcO)B]2O is exceptional in that C-B fission results in place of B-O fission. Due to the simplicity and ease of hydrolysis, this reaction is the best available method for conversion of the boronous to the boronic system. The failure of I to form complexes is attributed to back coordination from the O natom and strong +I effect of Bu groups rather than steric hindrance. The failure of Et2NH to cause B-O fission is in accord with previous work (cf. C.A. 51, 9476e); in contrast with the

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HOAc system, B-C fission does not take place. Prepns. are given.
    ANSWER 292 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN
L4
AN
     1958:108074 CAPLUS
     52:108074
DN
OREF 52:19108h-i
ΤI
    Additives for gasolines, lubricating oils, and electrical oils
    Airs, Raymond S.
IN
PA
     "Shell" Research Ltd.
DT
    Patent
LΑ
    Unavailable
FAN.CNT 1
                  KIND DATE
     PATENT NO.
                                        APPLICATION NO. DATE
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                                          _____
    GB 794380
PΤ
                           19580430
                                          GB
    DE 1060187
     2622-89-1, Borinic acid, diphenyl-
IT
        (esters with amino alcs., gasoline or oils contg.)
RN
     2622-89-1 CAPLUS
CN
    Borinic acid, diphenyl- (6CI, 7CI, 8CI, 9CI) (CA INDEX NAME)
   Ph
Ph-B-OH
AΒ
     Scavengers to remove engine deposits caused by leaded gasolines consist of
     an ester of an alkanolamine and a boronic or borinic acid with at least 3
    C atoms attached to B by a C-B bond. Specifically mentioned are
     3-azapentamethylene phenylboronate, m. 212.degree. (decomp.);
    bis(2-aminoethyl) phenylboronate, m. 128.degree.; 2-aminoethyl
     diphenylborinate, m. 189.degree. (from aq. methylated spirits);
    N-hexyl-3-azapentamethylene phenylboronate, m. 96-8.degree. (from petr.
     ether); and diphenyl borinic ester of Priminox 43. A B-content of about
     0.25 g./gal. is recommended. The additives are also useful as
     antioxidants in lubricants, elec. oils, fatty oils, and synthetic oils.
    ANSWER 293 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN
L4
AN
    1958:97646 CAPLUS
DN
     52:97646
OREF 52:17148f-i,17149a-b
TI
    Organoboron compounds. XXII. Mechanism of hydrolysis of esters of
     diarylboric acids
    Mikhailov, B. M.; Vaver, V. A.
ΑU
CS
    N. D. Zelinskii Inst. Org. Chem., Moscow
SO
     Izvestiya Akademii Nauk SSSR, Seriya Khimicheskaya (1958) 419-24
     CODEN: IASKA6; ISSN: 0002-3353
DT
     Journal
LΑ
    Unavailable
IT
     66117-64-4, Borinic acid, di-p-tolyl- 73774-44-4,
     Borinic acid, di-o-tolyl-
        (and their derivs.)
RN
     66117-64-4 CAPLUS
```

Borinic acid, bis(4-methylphenyl) - (9CI) (CA INDEX NAME)

CN

RN 73774-44-4 CAPLUS

CN Borinic acid, bis(2-methylphenyl) - (9CI) (CA INDEX NAME)

IT 2622-89-1, Borinic acid, diphenyl-

(esters and their derivs.)

RN 2622-89-1 CAPLUS

CN Borinic acid, diphenyl- (6CI, 7CI, 8CI, 9CI) (CA INDEX NAME)

IT 66117-64-4, Borinic acid, di-p-tolyl- 73774-44-4,

Borinic acid, di-o-tolyl-

(esters, and their derivs.)

RN 66117-64-4 CAPLUS

CN Borinic acid, bis(4-methylphenyl) - (9CI) (CA INDEX NAME)

RN 73774-44-4 CAPLUS

CN Borinic acid, bis(2-methylphenyl) - (9CI) (CA INDEX NAME)

AB cf. C.A. 50, 11964c; 52, 6237h. Hydrolysis of Ar2BOR by aq. NH4OH proceeds through formation of ammoniates of these esters with transformation to ammonium diarylborenates. Treatment of 1 mole PhMgBr at -50.degree. with 0.45 mole (BuO)3B and stirring 10 hrs. at -75.degree. gave after treatment with 5% HCl 46.7% Ph2BOBu, bl 110-20.degree., d20 0.9834, n20D 1.5471. Similarly (iso-AmO)3B gave 42% iso-AmOBPh2, b2 128-9.degree., d20 0.9710, n20D 1.5405, and 43.5% (iso-AmO)2BPh, b2 116-17.degree., d20 0.9221. Use of (PrO)3B similarly gave 27% Ph2BOPr, b5 125-6.degree., 0.9851, 1.5491, and 48.5% phenylboric anhydride, m. 187-9.degree.. All the above esters are slowly oxidized by air. (iso-BuO)3B and p-MeC6H4MgBr gave similarly 19.6% iso-BuOB(C6H4Me-p)2, b2.5 146-6.5.degree., 0.9630, 1.5448, and 30% p-tolylboric acid, m. 227-30.degree., and some (p-MeC6H4)3B, bl2 230-5.degree.. Use of .omicron.-MeC6H4MgBr gave some iso-BuOB(C6H4Me-.omicron.)2, bl.2 135.degree., 0.9704, 1.5440, along with 55% (iso-BuO)2BC6H4Me-.omicron., b3 110-12.degree., and 16.3% (.omicron.-MeC6H4)3B, b2 182-4.degree.. esters of diarylboric acids (1-2 g.) were treated in dry petr. ether with dry NH3 yielding the corresponding ammoniates, Ar2B(OR)NH3 (Ar and R shown, resp.): Ph, Br, m. 104-7.degree.; Ph, Bu, m. 97-9.degree.; Ph, iso-Bu (I), m. 103-5.degree.; Ph, iso-Am, m. 99-101.degree.; p-tolyl, iso-Bu, m. 77-9.degree.; .omicron.-tolyl, iso-Bu, m. 88-90.5.degree.. 5 ml. 30% aq. NH4OH was added 0.66 g. (p-MeC6H4)2BOCH2CHMe 2 and after shaking 10 min. the pptd. salt was sepd. yielding 86% (p-MeC6H4)2B(OH)2.NHs4 (II), m. 89-91.degree. (C6H6), insol. in H2O. Similarly was prepd. the .omicron.-tolyl analog, m. 97-100.degree.. Treatment of I with H2O at room temp. gave Ph2B(OH)2.NH4, m. 108-10.degree.. The other ammoniates hydrolyzed similarly. II in Et20 was treated with 1:2 HCl 5 min. yielding on evapn. of the org. layer 88% (p-MeC6H4)2BOH, m. 65-6.degree. (petr. ether). Similarly was prepd. the o-isomer, m. 64-6.degree. iso-BuOB(C6H4Me-p)2 in aq. EtOH was treated with H2NCH2CH2OH yielding a ppt. of (p-MeC6H4)2BOCH2CH2NH2, m. 174-6.degree.; similarly was prepd. the o-isomer, m. 179-80.5.degree.. (p-ClC6H4)2BOCH2CHMe2.NH3, m. 115-17.degree.; the p-bromo analog, m. 113-15.degree..

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L4 ANSWER 294 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN
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AN 1958:92778 CAPLUS

DN 52:92778

OREF 52:16310f-h

TI A study of the steric requirements of the 9-anthryl group and their contribution to the preparation of 9-anthrylboron compounds

AU Dodson, Vance H.; Fisher, William E.

CS Univ. of Toledo, Toledo, O.

SO Ohio Journal of Science (1958), 58, 141-4 CODEN: OJSCA9; ISSN: 0030-0950

DT Journal

LA Unavailable

RN 124108-68-5 CAPLUS

CN Borinic acid, di-9-anthryl- (6CI) (CA INDEX NAME)

AB Tri-9-anthrylborane(I) is not formed under reaction conditions more strenuous than those which lead to the easy formation of tri-.alpha.-naphthylborane and the more difficult formation of trimesitylborane which indicates that the steric factor in the reaction is not controlling as I should have the median crowding of the B atom of these 3 compds. It is possible that .pi.-electron repulsion on the 1 and 8 anthryl carbons may be the reason why only disubstitution of the B by aryls occurs here. Anthracene was converted to 9,10-dibromo-9,10dihydroanthracene which was dehydrohalogenated to 9-bromoanthracene, m. 99-100.degree.. With Mg-Et20 (II) gave 60-65% 9-anthrylmagnesium bromide (III). No I came from BF3.Et2O reaction with III; bright orange di-9-anthrylborinic acid (IV) was formed instead, m. 152.5.degree.. vacuo at 120 degree., IV disproportionates to anthracene and 9-anthrylboric oxide (V), orange-brown, m. 145.degree. to brown liquid. In air at 225.degree. IV oxidizes slowly to V and anthraquinone. IV yields 9-anthrylboronic acid with hot dil. HCl, disodio-9-anthrylboronate with hot dil. NaOH, and no hydrolytic product with boiling water. Di-9-anthryl boronfluoride formed intermediately in making IV was not isolated nor characterized.

L4 ANSWER 295 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN AN 1958:87916 CAPLUS DN 52:87916 OREF 52:15457g-h ΤI Dephenylation reactions of phenylboron acids and esters ΑU Abel, E. W.; Gerrard, W.; Lappert, M. F. CS Northern Polytechnic, London SO Journal of the Chemical Society, Abstracts (1958) 1451-3 CODEN: JCSAAZ; ISSN: 0590-9791 DT Journal LΑ Unavailable IT 2622-89-1, Borinic acid, diphenyl-(esters) RN 2622-89-1 CAPLUS CN Borinic acid, diphenyl- (6CI, 7CI, 8CI, 9CI) (CA INDEX NAME)

(prepn. of
AB PhB(OH)2 (I) 25 hrs. at 200.degree. gives C6H6 and HBO2. Ph2BOH (II) 8
hrs. at 175.degree. gives C6H6 and PhBO. II 20 hrs. at 175.degree. with
H2O gives C6H6 and H3BO3. C6H13CHMeOH (III) and excess C6H13CHMeOBPh2
(IV) 12 hrs. at 200.degree. slowly form C6H6 and (C6H13CHMeO)2BPh. When

approx. equal amts. of III and IV are used the product is (C6H13CHMeO)3B. Me and Bu esters react similarly with the corresponding alcs. C8H17OBPh2 (V) and BuOH 50 hrs. at 200.degree. give a mixt. of B(OBu)3 and B(OC8H17)3. EtOBPh2 100 hrs. at 200.degree. gives a mixt. of PhB(OEt)2, Ph3B, C6H6, and B(OEt)3. V 130 hrs. at 340.degree. gives octene, C6H6, and I. PhB(OMe)2 100 hrs. at 200.degree. gives Ph2B(OMe) and B(OMe)3. Ph2BOBu and B(OBu)3 100 hrs. at 200.degree. give some PhB(OBu)2.

T.4 ANSWER 296 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN AN 1958:87915 CAPLUS DN 52:87915 OREF 52:15456i,15457a-g The inductive effect of alkyl groups as determined by desilylation TI UΑ Benkeser, Robert A.; Hickner, Richard A.; Hoke, Donald I. CS Purdue Univ., Lafayette, IN Journal of the American Chemical Society (1958), 80, 2279-82 SO CODEN: JACSAT; ISSN: 0002-7863 DTJournal LΑ Unavailable ΙT 2622-89-1, Borinic acid, diphenyl-(prepn. of) RN2622-89-1 CAPLUS

Borinic acid, diphenyl- (6CI, 7CI, 8CI, 9CI) (CA INDEX NAME)

CN

AΒ The rates of the desilylation of m-alkyl-substituted trimethyl and triethylphenylsilanes increase in the order H < Me < Et < iso-Pr < Me3C, contrary to the decrease in rate for this series in the solvolysis of (m-alkylphenyl)dimethylcarbinyl chlorides. It is rationalized that, at least in this instance, the desilylation reactions more closely approximate conditions realized in true aromatic substitutions. carbinyl chloride series is apparently complicated by unusual hyper-conjugative or steric effects. EtMgBr from 109 g. EtBr and 26 g. Mg in 250 cc. dry Et20 added with stirring to 157 g. Et2SiCl2, b. 128.degree., in 250 cc. dry Et2O, filtered, and distd. yielded 89 g. Et3SiCl (I), b. 137-49.degree., n20D 1.4340. m-ClC6H4MgBr from 7.3 g. Mg in 50 cc. Et20 and 57.5 g. m-ClC6H4Br in 150 cc. Et20 treated with 17.4 q. Me2CO at such a rate as to maintain gentle reflux, stirred 15 min. at room temp., poured onto chipped ice, and worked up in the usual manner yielded 34.8 g. m-ClC6H4C(OH)Me2 (II), b2.5 86-8.degree., n20D 1.5380. II (51 g.), 2 g. KHSO4, and 0.5 g. hydroquinone distd. during 40 min., and the distillate, b95 12530.degree., dissolved in Et2O, washed, dried, and distd. yielded 29 g. m-ClC6H4CMe:CH2 (III), b4 60-2.degree., n20D 1.5536, and 6.4 g. crude II. III (35.8 g.), 50 cc. abs. EtOH, and 0.3 g. 10% Pd-C hydrogenated about 2 hrs. at an initial pressure of 55 lb. yielded 24.8 q. m-ClC6H4CHMe2, b8 66-8.degree., n20D 1.5136. By standard methods was prepd. m-ClC6H4CMe3, bl1 88.degree., n20D 1.5127. The appropriate m-chloro or m-bromoalkylbenzene and 0.1 mole Me3SiCl or Et3SiCl in 50 cc. dry Et20 added during 15 min. with stirring to 6.5 g. Na sand in 50. cc. dry Et20 under N, the mixt. refluxed with stirring until most of the Na had reacted, treated with cooling dropwise with 100 cc. H2O, and worked up in the usual manner gave the corresponding m-(trialkylsilyl)alkylbenzenes.

In this manner were prepd. the following compds. m-RC6H4SiMe3 (IV) (R, n20D, d20, b.p./mm., MRD, and % yield given): H, 1.4910, -, 169.degree./760, -, 65; Me, 1.4924, -, 190.degree./760, -, 55; Et, 1.4924, 0.8645, 206-7.degree./760, 59.88, 50; iso-Pr, 1.4894, 0.8594, 0 8597, 94.degree./14, 75; Me3C, 1.4884, 0.8617 82.degree./6, 55, m-RC6H4SiEt3 (V) (same data given): Me, 1.5022, -, 123.degree./11, -, 24; Et, 1.5015 0.8833, 125.degree./9, 73.58, 20; iso-Pr, 1.4972, 0.8902, 131.degree./9, 78.03, 15; Me3C, 1.4956, 0.8747, 145.degree./11, 82.93, 26. An aliquot of a stock soln. of aq. HCl in glacial AcOH dild. to about 35 cc., allowed to stand overnight, added to 0.3-0.5 g. of the appropriate silane, the mixt. dild. to 50.0 cc. with glacial AcOH, and the expansion of the mixt. measured in a dilatometer gave the data for the detn. of the pseudo 1st-order rate const. of the cleavage reaction. In this manner were detd. the av. rate consts. and half-lives for the acid-catalyzed cleavage of the following IV in glacial AcOH (2.35M in HCl and 7.23M in H2O) at 25.degree. (R of IV, av. k .times. 103 min.-1 and half-life in min. given): H, 3.84, 181; Me, 8.31, 83; Et, 8.54, 81; iso-Pr, 9.08, 71; Me3C, 10.8, 64. The same data were detd. for the following compds. V (same data given): H, 1.64, 422; Me, 3.87, 179; Et, 4.69, 1.48; iso-Br, 5.14, 135; Me3C, 5.90, 117. By standard methods were prepd. the following compds.: Et3SiPh, b9 105.degree., n20D 1.5024; m-Br-C6H4Ac, b4 96-7.degree., n20D 1.5759; m-BrC6H4Et, b20 91.degree..

L4 ANSWER 297 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

AN 1958:25307 CAPLUS

DN 52:25307

OREF 52:4532f-h

TI Organoboron compounds. XVIII. New method of synthesis of diarylborinic acids

AU Mikhailov, B. M.; Vaver, V. A.

CS N. D. Zelinskii Inst. Org. Chem. Acad. Sci. U.S.S.R., Moscow

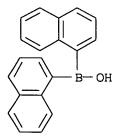
SO Izvestiya Akademii Nauk SSSR, Seriya Khimicheskaya (1957) 989-91 CODEN: IASKA6; ISSN: 0002-3353

DT Journal

LA Unavailable

RN 62981-91-3 CAPLUS

CN Borinic acid, di-1-naphthalenyl- (9CI) (CA INDEX NAME)



AB cf. C.A. 51, 3487c; 52, 3667h. To 0.2 mole PhMgBr was added with cooling 9 g. (iso-BuOBO)3 in C6H6, the mixt. refluxed 1 hr. and treated with 200 ml. 3.5% HCl, and the org. layer, sepd. rapidly and evapd. rapidly in vacuo with min. heating, yielding on addn. of Et2O-hexane to the residue 8.3 g. phenylboronic anhydride, m. 190-3.degree., while the mother liquor after evapn. and addn. of isopentane gave 51.6% (Ph2B)2O, m.

130-1.degree.. The combined distillates gave iso-BuOH and some H3BO3. PhMgBr treated as above with (C6H110BO)3 and the crude product treated with HOCH2CH2NH2 gave 62% Ph2BOCH2CH2NH2, m. 190-1.degree. (EtOH). (iso-BuOBO)3 with 1-C10H7MgBr gave similarly after treatment with 3.5% HCl 76.6% (1-C10H7)2BOH, m. 114-15.degree.. Similar reaction of p-MeC6H4MgBr and treatment of the crude product with HOCH2CH2NH2 gave 41% (p-MeC6H4)2BOCH2CH2NH2, m. 174.5-76.degree. (EtOH), along with 21.2% p-MeC6H4B(OCH2CHMe2)2, b5.5 127-8.degree., d20 0.9106, n20D 1.4761. The use of 0.2 mole .omicron.-MeC6H4MgBr and 0.03 mole (iso-BuOBO)3 gave after esterification of crude products with iso-BuOH 21% .omicron.-MeC6H4B(OCH2CHMe2)2, b3 111-15.degree., and 42% (.omicron.-MeC6H4B)2BOCH2CHMe2, b2.5 151-3.degree., 0.9701, 1.5432. The latter ester is slowly oxidized by contact with air.

L4 ANSWER 298 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

AN 1957:85554 CAPLUS

DN 51:85554

OREF 51:15440b-f

TI Organoboron compounds. XV. Organoboron compounds with an asymmetric boron atom

AU Mikhailov, B. M.; Kostroma, T. V.; Fedotov, N. S.

CS N. D. Zelinskii Inst. Org. Chem., Moscow

SO Izvest. Akad. Nauk S.S.S.R., Otdel. Khim. Nauk (1957) 589-97

DT Journal

LA Unavailable

RN 109846-74-4 CAPLUS

CN Borinic acid, phenyl-o-tolyl- (6CI) (CA INDEX NAME)

RN 124422-00-0 CAPLUS

CN Borinic acid, (p-chlorophenyl)phenyl- (6CI) (CA INDEX NAME)

AB cf. C.A. 50, 11964b; 51, 1882h, 8675d. Addn. to 19.6 g. PhB(OCH2CHMe2)Cl in Et2O of o-MeC6H4MgBr from 20.5 g. RBr gave after removal of Et2O, addn. of isopentane, filtration, and distn. 62.1% o-MeC6H4BPhOCH2 CHMe2 (I), b9.5 156-9.degree., d20 0.9714 nD20 1.5370; similarly, p-BrC6H4MgBr gave 34.7% p-BrC6H4(o-MeC6H4)BOCH2CHMe2, b.p. unstated, 1.1758, 1.5615. Similarly, p-ClC6H4MgBr gave 44.4% p-ClC6H4BPhOCH2CHMe2 (II), b2

137-9.degree., 1.0351, 1.5432. I (26 g.) treated with 23.5 g. PCl5 gave 67.2% o-MeC6H4BPhCl (IIa), b7 143-6.degree., d20 1.090; this treated with H2O, extd. with isopentane, and thoroughly dried in vacuo gave 84.2% o-MeC6H4B(OH)Ph.H2O, a liquid, which on standing gradually formed the anhydride of phenylboronic acid (III). Similarly, II gave 47% p-ClC6H4BPhCl, b7 135-40.degree., d20 1.145, which with H2O gave 76% p-ClC6H4B(OH)Ph.H2O, liquid, gradually changing to III on standing. Ph2BCl and o-MeC6H4MgBr gave in 7 hrs. 49% o-MeC6H4BPh2, b3 167-9.degree., which oxidizes in air. Similarly, IIa and p-MeC6H4MgBr gave (o-MeC6H4)BPhC6H4Me-p (IV), 48.4%, b2 168-70.degree.. Similarly was prepd. 49% o-MeC6H4B(C6H4Cl-p)Ph (V), b2 163-7.degree., slowly solidifying on standing. Refluxing 10 g. Ph2BCl with o-MeC6H4Li from 11 g. RBr 6 hrs. in Et20 gave 2.8 g. (o-MeC6H4)2BPh2Li and 4.7 g. o-MeC6H4BPh2, b4 177-80.degree.. The latter with 1-Cl0H7Li in Et2O gave 49% [1-C10H7BPh2C6H4M-e-o]Li, a solid, isolated as an adduct with 2 mols. This with satd. KCl gave [1-C10H7BPh2C6H4Me-o]K, colorless solid. IV similarly gave [o-MeC6H4(p-MeC6H4)BPhC10H7-1]Li.2Et2O, which with KCl yielded the K salt, C30H26BK. V and 1-C10H7Li similarly gave 39% [o-MeC6H4(p-ClC6H4)BPhC10H7-1]Li.2-Et2O, solid, yielding with KCl the K salt, C29H23KClB, cryst. powder.

ANSWER 299 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN L4

1957:71312 CAPLUS AN

51:71312 DN

OREF 51:12843h-i,12844a-h

Studies in stereochemistry. XXII. The preparation and reactions of ΤI trimesitylborane. Evidence for the nonlocalized nature of the odd electron in triarylborane radical ions and related free radicals

ΑU Brown, Herbert C.; Dodson, Vance H.

CS

Purdue Univ., Lafayette, IN J. Am. Chem. Soc. (1957), 79, 2302-6 SO CODEN: JACSAT; ISSN: 0002-7863

DTJournal

LΑ Unavailable

IT 20631-84-9, Borinic acid, dimesityl- 62981-91-3, Borinic acid, di-1-naphthyl-(prepn. of)

RN 20631-84-9 CAPLUS

CN Borinic acid, bis(2,4,6-trimethylphenyl)- (9CI) (CA INDEX NAME)

62981-91-3 CAPLUS RN

Borinic acid, di-1-naphthalenyl- (9CI) (CA INDEX NAME) CN

AB cf. C.A. 48, 557i. Ph3B (I) white crystals, m. 138.degree. (under N), was prepd. by the method of Krause and Nitsche (C.A. 16, 3640) and stored in sealed tubes under N. (1-C10H7)3B (II), m. 205-6.degree. (from C6H6), was prepd. by the method described previously (C.A. 42, 8790q) from 1-C10H7Br, b20 146-8.degree., and dried in a stream of N. 2,4,6-Me3C6H2Br (47.8 q.) in dry Et20 added dropwise to 7.0 g. Mg and 100 cc. dry Et20 during 1 hr. at reflux temp., the mixt. refluxed 4-5 hrs. under a stream of N, kept at room temp. overnight under N, dild. with 150 cc. dry PhMe, refluxed 0.5 hr., treated with stirring at reflux temp. dropwise with 5.1 g. Et20.BF3 in 50 cc. Et20, dild. with 50 cc. PhMe after removal of the Et20, refluxed 4 hrs. with stirring, cooled to room temp., poured onto 300 cc. crushed ice and H2O, treated with 55 cc. concd. HCl and occasionally with ice, and shaken 20 min., the org. layer washed with three 200-cc. portions cold H2O, dried, concd. to 1/2 vol., dild. with 150 cc. 95% EtOH, and filtered, and the ppt. recrystd. from 300 cc. 95% EtOH yielded 4.5 g. (2,4,6-Me3C6H2)3B (III), m. 190.5-1.5.degree.; 2nd crop, about 2 g. Soly. of III in 100 cc. solvent at 25.degree.: 6.96 g. CCl4, 1.25 g. Me2CO, 0.08 g. 95% EtOH, 4.98 g. Et2O, 16.5 g. C6H6: 2,4,6-Me3C6H2MgBr (0.228 mole) treated during 1.5 hrs. with 0.073 mole Et20.BF3, the mixt. refluxed 2 hrs., and kept 24 hrs. at room temp., the orange Et20 layer evapd., and the residue distd. yielded (2,4,6-Me3C6H2)2BF (IV), b0.5 127-9.degree., m. 75.5-6.0.degree. (sealed tube). IV treated with H2O gave (2,4,6-Me3C6H2)2BOH, m. 140-1.degree. (from petr. ether). I and II in dry Et20 satd. with dry NH3 and evapd. gave I.NH3 and II.NH3, resp. not react with NH3 under the same conditions. I and II in dry C6H6 could be titrated with standard NaOMe and NaOCMe3 and phenolphthalein, while the 1st drop of alkoxide soln. added to III in C6H6 gave a pink color. A titrated soln. of II in C6H6 evapd. in vacuo left II.NaOMe white solid which turned brown at 155-60.degree. and black at 200.degree. without melting; C6H6 solns. of the salt shaken with 50 cc. H2O and titrated with HCl indicated a 1:1 mole ratio of II and NaOMe; as hydrolysis products were identified C10H8 and (1-C10H7)2BOH, m. 148-50.degree.. II.-NaOMe in C6H6 treated with dry HCl, heated until all HCl was removed, filtered, and evapd. to dryness gave II, m. 204-6.degree. I (0.933 millimole) dissolved in 20.0 cc. 1-C10H7Br, evapd. to 0.5 mm., and kept under O showed the following consumption of O (time in hrs. and cc. O reacted are given): 0.25, 9.9; 0.50, 11.5; 0.75, 11.6; 1.25, 12.4; 2.25, 12.4; 24.0, 12.4. II (0.496 millimole) was treated in the same manner (time in days and cc. O reacted): 3, 7.9; 17, 11.9; 42, 12.8; 54, 13.2; 66, 13.1. did not show O absorption under the same conditions during 15 months. Solid I and H2O shaken intermittently during 8 hrs. at room temp. and filtered, and the pale brown residue dried in air and recrystd. from petr. ether gave PhBO, m. 201.5-2.5.degree., the aq. soln. evapd. gave a small amt. of PhB(OH)2, m. 215.degree.. II did not react with H2O at room temp. during 8 hrs., but refluxing with H2O during 2.5 hrs. gave complete conversion to C10H8 and 1-C10H7B(OH)2, m. 195.degree.. III did not react with H2O at reflux temp. during 8 hrs. I shaken 4 hrs. at room temp. with

0.05N NaOH, the resulting soln. neutralized with dil. HCl and extd. with Et2O, and the ext. worked up gave PhB(OH)2, m. 215.degree.. II gave with 0.05N NaOH at room temp. during 4 days a ppt. of C10H8 and unreacted II; the aq. filtrate neutralized and extd. with Et2O yielded a small amt. of 1-C10H7B(OH)2, m. 195.degree.. III did not react with 0.05N NaOH during 4 days at room temp. A weighed sample of III and Na amalgam under dry N treated with dry Et2O, sealed in a bulb, and kept at room temp. with occasional shaking for 24 hrs., the mixt. rinsed with Et2O into H2O, the Et2O removed, and the aq. mixt. contg. a white ppt. titrated with standard HCl and filtered gave unchanged III; the base was assumed to be NaOH; the mole ratio of Na to III varied from 1.09:1 to 1.53: 1; in a similar run the ratio was 1.9:1 after 2 months. I and II reacted with similar ease under the same conditions.

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ANSWER 300 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN
T.4
AN
     1957:51703 CAPLUS
DN
     51:51703
OREF 51:9513c-i
     Preparation of esters of diphenylboronous acid
TI
ΑU
     Abel, E. W.; Gerrard, W.; Lappert, M. F.
CS
     Northern Polytech., London
SO
     J. Chem. Soc. (1957) 112-15
DT
     Journal
LΑ
     Unavailable
ΙT
     2622-89-1, Borinic acid, diphenyl-
        (esters)
RN
     2622-89-1 CAPLUS
CN
     Borinic acid, diphenyl- (6CI, 7CI, 8CI, 9CI) (CA INDEX NAME)
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AB Ph2BOH (I) and Ph2BOBPh2 (II) directly esterified by primary and secondary alcs. and PhOH gave alkyl and Ph diphenylboronites. Higher homologs were also obtained by alcoholysis of a lower member of the series. The procedure of widest application involved interaction of Ph2BCl (III) with the alc., and was suitable for the prepn. of Me and tert-Bu homologs. methods were discussed. The esterification of I was carried out on a 0.05-molar scale. I or II heated with the appropriate alc., sufficient alc. used to remove H2O formed as an azeotrope, for lower alcs., this carried out at normal pressure, but for PhOH and higher alcs. at 20 mm., and the esters distd. under reduced pressure under N gave the following results (R in Ph2BOR, % yield, b.p./mm., d20, and nD20 given): Et, 80, 138.degree./10, 1.005, 1.5557; Pr, 91, 152.degree./10, 0.987, 1.5458; Bu, 93, 158.degree./9, 0.977, 1.5390; iso-Bu, 84, 153.degree./10, 0.963, 1.5276; sec-Bu, 86, 152.degree./10, 0.961, 1.5291; C8H17, 79, 155.degree./0.05, 0.950, 1.5269; 1-methylheptyl, 84, 148.degree./0.2, 0.941, 1.5196; neopentyl, 82, 114.degree./0.5, 0.965, 1.5370; Ph, 72, 140.degree./0.05, 1.084, 1.6053. The alcoholysis of Ph2BOR was carried out at 0.05 molar scale, 3 moles of the higher boiling alcs. added to 1mole Ph2BOR, the mixt. slowly fractionated and the crude esters distd. at reduced pressure in N. The results were as follows (R' in Ph2BOR', R''OH, % yield Ph2BOR'', b.p./mm., and nD20 given): Et, BuOH, 89, 158.degree./9, 1.5383; Bu, C8H17OH, 83, 160.degree./0.3, 1.5247; Pr, C6H13CHMeOH, 92, 152.degree./0.5, 1.5198 ([.alpha.]D20 -4.17); Bu, C6H13CHMeOH, 83,

140.degree./0.05, 1.5189 ([.alpha.]D20 -4.14.degree.); Et, tert-BuCH2OH, 85, 110.degree./0.2, 1.5369; Et, PhOH, 88, 139.degree./0.05, 1.6050. III was treated with alcs. on a 0.05-molar scale. The alc. (1 mole) in 20 cc. CH2Cl2 was added during 0.5 hr. to 1 mole III in 20 cc. CH2Cl2, HCl evolved and the solvent removed in vacuo, and the crude ester distd. under N. The following results were obtained (R in Ph2BOR, % yield, b.p./mm., nD20, and dD20 given): Me, 80, 132.degree./10, 1.5709, 1.032; tert-Bu, 57, 142.degree./10, 1.5396, 0.972; C6H13CHMe, 93, 134.degree./0.001, 1.5218, - ([.alpha.]D20 -4.20.degree.); tert-BuCH2, 79, 116.degree./0.5, 1.5374, -; Ph, 83, 136.degree./0.01, 1.6062, -. III (22.23 g.) added during 15 min. to 8.23 g. tert-BuOH at 0.degree. gave no evolution of gas but formed a white ppt.; tert-BuCl (7.9 g.) was removed and trapped at -80.degree., b. 53.degree., nD20 1.3858. The residue gave 18.25 g. II, m. 114.degree.. Reversal the of order of addn. gave the same results. III (2.68 g.) added during 15 min. at 0.degree. to 0.994 g. sec-BuOH without solvent gave evolution of HCl and 2.75 g. sec-Bu diphenylboronite, b8 149.degree., nD20 1.5310. Mol. refractivities were detd. for a series of Ph2BOR. The agreement between calcd. and found values confirmed the value (Torssell, C.A. 49, 10213f) of 4.39 detd. for B in this type of compd.

L4ANSWER 301 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN AN 1957:17149 CAPLUS 51:17149 DN OREF 51:3517e-h Aromatic boron compounds. V ΤI ΑU Neu, Richard CS Willmar Schwabe Firm, Karlsruhe, Germany SO Chem. Ber. (1955), 88, 1761-5 DT Journal LΑ Unavailable IT 66117-64-4, Borinic acid, di-p-tolyl-(prepn. of) ŔŊ 66117-64-4 CAPLUS CN Borinic acid, bis(4-methylphenyl) - (9CI) (CA INDEX NAME)

cf. C.A. 50, 17488f. The prepn. of diphenylborinic anhydride, (Ph2B)20, (I) and di-p-tolylborinic anhydride (II) are described. Boric oxide (70 g.) and 350 g. of BuOH gave 238 g. of tributyl borate (III), b17 118-19.degree. PhMgBr (from 2.4 g. of Mg and 20 g. of PhBr) in Et2O treated (ice-salt bath) with 40 g. III in Et2O gave a mixt. which after 0.5 hr. was hydrolyzed with a little ice and acidified with 3% HCl. The aq. phase was extd. with Et2O and the combined exts., with the Et2O layer from the reaction, were extd. with 2N NaOH. The residual Et2O soin. gave 2.3 g. of biphenyl. The cooled alk. exts. were acidified with 2N H2SO4 and extd. with petr. ether (b. 44-5.degree.). The solvent was evapd. from the H2O-washed and dried exts. and the residue set aside for 2 days to give crystals which were filtered off with suction and washed with cold petr. ether. The filtrates were stripped of solvent in vacuo and steam distd. (the addn. of BaCO3 is helpful). Extn. of the distillate with

petr. ether and evapn. of the solvent from the exts. gave 1.44 g. I, m. 112-13.degree. Using a better grade of Mg a yield of 16.2% of I was obtained. Similarly, 20% and 18.4% yields of II were obtained, m. 105-6.degree. One expt. with p-MeC6H4MgBr gave a substance, m. 38-40.degree., which may be di-p-tolylborinic acid (IV) and which is converted to II by drying over H2SO4 in a vacuum desiccator. II and IV gave di-p-tolylmercury in almost quant. yield when treated with an aq. MeOH soln. of HgCl2 on a small scale.

L4 ANSWER 302 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

AN 1956:64336 CAPLUS

DN 50:64336

OREF 50:11964c-h

TI Organoboron compounds. VIII. Synthesis and properties of diarylboric acids

AU Mikhailov, B. M.; Vaver, V. A.

CS N. D. Zelinskii Inst. Org. Chem., Acad. Sci. U.S.S.R., Moscow

SO Izvestiya Akademii Nauk SSSR, Seriya Khimicheskaya (1956) 451-6 CODEN: IASKA6; ISSN: 0002-3353

DT Journal

LA Unavailable

RN 62981-91-3 CAPLUS

CN Borinic acid, di-1-naphthalenyl- (9CI) (CA INDEX NAME)

RN 89566-59-6 CAPLUS

CN Borinic acid, bis(4-chlorophenyl) - (9CI) (CA INDEX NAME)

RN 96484-29-6 CAPLUS

CN Borinic acid, bis(4-bromophenyl) - (9CI) (CA INDEX NAME)

AΒ To 0.5 mole 1-C10H6MgBr was added at -30.degree. 0.225 mole (iso-BuO)3B and after 6-7 hrs. at -70.degree., the mixt. treated with 5% HCl, yielding on concn. of the org. layer 63% (1-C10H7)2BOBu-iso (I), m. 104-5.degree. (from hexane), after washing the residue with pentane; the wash liquid gave 15% 1-C10H7B(OBu-iso)2, b6 166-8.degree., d20 0.9777, which is hydrolyzed by atm. moisture. To 10 g. I in 20 ml. MeOH was added 15 ml. 30% NH4OH, yielding after 0.5 hr. 97.5% (1-C10H7)2B(OH)2NH4, m. 107-8.degree. (from MeOH), which decomposes rapidly in air yielding NH3, C10H8, and 1-C10H7BO2H2. If this NH4 salt in Et2O was stirred with 1:1 HCl and the org. layer evapd. after 0.5 hr., there was obtained 71.7% (1-C10H7)2BOH, m. 114.5-15.degree. (from petr. ether), which heated in vacuo gave C10H8 and 1-C10H7B(OH)2. The above acid (2 g.) refluxed 2 hrs. with 5 ml. SOCl2, concd. in vacuo and treated with C6H6-petr. ether, gave 98% [(1-C10H7)2B]20, m. 190-2.degree.. To 0.65 mole p-BrC6H4MqBr (cf. Pink, C.A. 18, 669) was added over 0.5 hr. at -30.degree. 0.25 mole (iso-BuO)3B in Et2O and after 8 hrs. at -70.degree. the mixt. was treated with 5% HCl, the org. layer concd. and the residue esterified with iso-BuOH; distn. gave 39% (p-BrC6H4)2BOBu-iso, (II) b1 161-3.degree., and 37% p-BrC6H4B(OBu-iso)2, bl 109-10.degree., d20 1.1583. II (2.25 g.) shaken with 5 ml. 30% NH4OH gave 87.3% (p-BrC6H4)2B(OH)2NH4, m. 134-5.degree. (from C6H6). II (1.23 g.) in 3.65 ml. 0.8N KOH was slowly distd. in vacuo at 70.degree. under N giving a residue of 0.8 g. (p-BrC6H4)2B(OH)2K, crystals (from C6H6-MeOH). The NH4 salt above with dil. HCl gave 86% (p-BrC6H4)2BOH, m. 90-1.degree. (from petr. ether), which forms similarly from the above K salt. Similarly, 0.6 mole p-ClC6H4MgBr and 0.25 mole (iso-BuO)3B gave 40% (p-ClC6H4)2BOBu-iso, (III), b1 134-5.degree., d20 1.1414, and 25% p-ClC6H4B(OBu-iso)2, b1 93-5.degree., d20 1.0051; the former is rapidly oxidized in air, the latter is immediately hydrolyzed in moist air. III shaken with satd. Ba(OH)2 gave 89.5% (p-ClC6H4)2B(OH)2Ba, insol. in H2O, sol. in MeOH and EtOH. Shaken with 1:4 HCl it gave (p-ClC6H4)2B(OH2, m. 76-8.degree. (from aq. EtOH); it is decompd. by drying in vacuo, yielding PhCl and p-ClC6H4B(OH)2. III with N NaOH gave (p-ClC6H4)2B(OH)2Na, which on acidification gave an acid, m. 70-3.degree., corresponding to (p-ClC6H4)2BOH.H2O. Cf. Konig and Scharrnbeck (C.A. 25, 927); Mel'nikov and Rokitskaya (C.A. 33, 4969.9).

L4 ANSWER 303 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

AN 1956:27736 CAPLUS

DN 50:27736

OREF 50:5551i,5552a-i

TI Organoboron compounds. V. The preparation of an unsymmetrical diarylborinate

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CS Northwestern Univ., Evanston, IL

SO Journal of the American Chemical Society (1955), 77, 2489-91 CODEN: JACSAT; ISSN: 0002-7863

DT Journal

LA Unavailable

IT 62981-91-3, Borinic acid, di-1-naphthyl-

(and esters)
RN 62981-91-3 CAPLUS

CN Borinic acid, di-1-naphthalenyl- (9CI) (CA INDEX NAME)

RN 13331-25-4 CAPLUS

CN Borinic acid, 1-naphthalenylphenyl- (9CI) (CA INDEX NAME)

AΒ The prepn. and some properties of Ph(1-C10H7)BO(CH2)2N2 (XI) and (1-C10H7)2BO(CH2)2NH2 (XII) are described. PhMgBr (1.05 moles) in 500 cc. Et20 added dropwise during 1.5 hrs. with stirring to 115 g. II in Et20 at Dry Ice temp., the mixt. left 10 hrs. to warm to room temp., hydrolyzed with dil. HCl, the Et20 layer mixed with 250 cc. 75% Et0H and 42.7 g. V, the mixt. warmed a short time on the steam bath, and the ppt. (109 g.) recrystd. from aq. EtOH yielded 64 g. I, fine white needles, m. 190-2.degree.. 1-C10H7Br (207 g.) in 350 cc. Et2O added to 29.2 g. Mg in 500 cc. Et2O, the mixt. dild. with 300 cc. C6H6, added during 5 hrs. to 125 g. II in 300 cc. Et2O at about -60.degree., allowed to stand overnight, hydrolyzed, the org. layer treated with cooling with 42.7 g. V, and the ppt. washed with 200 cc. warm 50% aq. EtOH gave 94.5 g. XII, m. 205-6.degree., purified by liberating the acid with Et20 and HCl and treating the Et2O layer with V. In 1 run was obtained a sample of XII, m. 191-3.degree., whose m.p. gradually increased during several weeks to 205-6.degree.; both samples became tan-colored after a few months in air. II warmed with 50% aq. EtOH-HCl, the soln. poured into ice water, and the ppt. recrystd. and dried gave (1-C10H7)2BOH (XIII), m. 105-6.degree... XIII treated with V in aq. EtOH gave 74% XII. XIII (0.50 g.) heated 6 hrs. under N at 120-30.degree. gave 0.22 g. C10H8, m. 80-1.degree.; the residue dissolved in Et20, extd. with 10% aq. NaOH, and acidified yielded 0.2 g. 1-C10H7B(OH)2, m. 194-6.degree.. 1-C10H7MgBr (0.049 mole) in about 20 cc. Et20 and 3 cc. C6H6 added dropwise at -60.degree. to 11.70 q. PhB(OBu)2 (XIV), b2 102-5.degree., in 100 cc. Et2O, the mixt. kept at room temp. overnight, hydrolyzed with dil. HCl, the Et20 layer evapd., the residue treated with 125 cc. EtOH, 20 cc. H2O, and then 6 cc. V, and the ppt. (10.1 q.) recrystd. from ag. Me2CO gave XI, m. 228-9.degree..

1-C10H7MgBr (0.146 mole) and 0.15 mole XIV gave in a similar run 88% XI, m. 221-3.degree.. 1-C10H7B(OH)2 distd. with BuOH gave the di-Bu ester, b1 170-4.degree., nD27 1.5322; this ester (28.4 g.) treated with 392 co. 0.097N PhMgBr in Et20; and the mixt. worked up in the usual manner gave 24 g. XI, m. 221-2.degree.. XI (1.5 g.) shaken with Et2O and 6N HCl, the Et20 layer evapd. on the steam bath, the residue dild. with 20 cc. EtOH, then almost to turbidity with H2O, treated with 1 cc. Me2C(NH2)CH2OH in 1 cc. EtOH, filtered after 10 min., and the filter cake washed with EtOH and Et2O and dried gave 1.43 g. Ph(1-C10H7)BOCH2C(NH2)Me2 (XV), m. 199-200 degree.; the filtrate from the XV dild. with H2O gave an addnl. 0.082 g. XV, m. 195-7.degree.. (1-C10H7)2BOCH2C(NH2)Me2, m. 208-10.degree., was prepd. similarly from XII. XI (0.500 g.) shaken with Et20 and 6N HCl, the Et20 layer evapd., the residue treated with 2 cc. Me2N(CH2)2OH in 2 cc. 95% EtOH, and the mixt. warmed 10 min. on the steam bath, dild. during 5 min. (on the steam bath) with 25 cc. H2O, cooled, and filtered gave 0.200 g. C10H8, m. 79-80.degree.; the filtrate acidified and extd. with Et20 gave 0.181 g. PhB(OH)2, m. 209-15.degree.. XI (0.500 g.), treated in the same manner but at room temp., gave 19% C10H8. XI (0.500 g.) heated 10 min. on the steam bath with 2 cc. V and 2 cc. EtOH and the mixt. cooled and filtered gave 0.471 g. unchanged XI. XI (4.99 g.) treated with 25 cc. 3.3M AcOH contg. 3.2 g. NaF and 4.92 g. NaOAc, the mixt. treated during 2 hrs. with stirring and refluxing with 16.0 g. Br and 14.4 g. KBr in 50 cc. 3.3M AcOH, refluxed 0.5 hr., cooled, treated with NaHSO3, basified, steam distd. during 0.5 hr., and the distillate dissolved in 20 cc. pentane and 3 cc. Et20, dried, and distd. gave 1.92. g. PhBr, b. 149-64.degree., nD27.4 1.5528; the higher-boiling residue (1.08 g.) combined with the org. material obtained by steam distg. the original reaction mixt. an addnl. 0.5 hr. and redistd. gave 1.8 g. 1-C10H7Br, b27 153-6.degree., nD30 1.6442; the residue recrystd. gave 0.17 g. 1,4-C10H6Br2, m. 74-6.degree.. XI (0.5 g.) in 10 cc. 50% EtOH treated with 5 cc. 30% H2O2 gave 0.1 g. 1-Cl0H7OH, m. 92.degree.. XI (1 g.) and 2 g. ZnCl2 in 20 cc. H2O steam distd. gave 0.28 g. C10H8; in the absence of the ZnCl2 the cleavage was very slow. XI in dil. HCl steam distd. gave only a trace of C10H8; the soln. basified gave 75% unchanged XI.

L4 ANSWER 304 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

AN 1956:27735 CAPLUS

DN 50:27735

OREF 50:5550i,5551a-i

TI Organoboron compounds. IV. Aminoethyl diarylborinates

AU Letsinger, Robert L.; Skoog, Ivan

CS Northwestern Univ., Evanston, IL

SO Journal of the American Chemical Society (1955), 77, 2491-4 CODEN: JACSAT; ISSN: 0002-7863

DT Journal

LA Unavailable

RN 96484-29-6 CAPLUS

CN Borinic acid, bis(4-bromophenyl) - (9CI) (CA INDEX NAME)

GΙ For diagram(s), see printed CA Issue.

AΒ cf. C.A. 49, 8840d. A practical procedure has been developed for the prepn. of Ph2BO(CH2)2NH2 (I); this involves the reaction of PhMgBr with (BuO)3B (II) at low temp., isolation of Ph2BOBu (III) as a complex (IV) with NH3, and conversion of the IV to I with H2N(CH2)2OH (V). (p-BrC6H4)2BO(CH2)2NH2 (VI) was prepd. analogously. Solid derivs. of arylboronic acids may be obtained by esterifying them with (HOCH2CH2)2NH (iso-BuO)3B (60.2 g.) in 150 cc. Et2O treated at room temp. slowly with stirring with 475 cc. 1.2M PhMqBr under N, the mixt. refluxed 1 hr., stirred 2 hrs. with heating, hydrolyzed with 200 cc. 3.6M HCl, and the Et20 layer washed, dried, and distd. gave the following fractions (b15 and wt. in g. given): 93-150.degree., 1.15; 150-5.degree., 2.00; 155-60.degree., 2.70; 160-9.degree., 16.60; 169-75.degree., 7.25; 175-205.degree., - (mainly Ph3B); fraction 2 crystd. in the condenser and on recrystn. gave 0.62 g. Ph2, m. 67-8.degree.; fractions 3-5 were liquids and 6 was solid. All portions dild. with an equal vol. of Et20 and satd. with dry NH3 gave ppts.; fraction 2 gave, after removal of the Ph2, 0.75 g. ppt.; the total yield from 2-5 was 16.9 g. Ph2BOCH2CHMe2 (VIII)-NH3 complex, m. 64-7.degree. with evolution of NH3; it decompd. rapidly in C6H6 at about 70.degree.; after standing several weeks it became slightly discolored (iso-BuOH odor). Ph3B.NH3 decompd. in air at about 170-6.degree.. PhB(OBu)2 and PhB(OPr)2 could not be pptd. from Et2O with NH3; the Et2O soln. evapd. however, gave a white solid which decompd. with evolution of NH3 at about 30.degree.. PhMgBr (0.394 mole) in 420 cc. Et20 added very slowly to 45.6 g. II in 500 cc. Et20 at about -60.degree., the soln. kept at room temp. overnight, treated with dil. HCl, the Et2O layer distd., and the residue distd. with 10 cc. BuOH and 700 cc. PhMe gave the following fractions: 10.45 g., b1 70-125.degree., 28.52 g., b1 125 37.degree., and 3.08 g., bl 137-65.degree.; each fraction dild. with an equal vol. of Et2O and satd. with NH3 gave 2.34, 22.56, and 0.53 g. NH3 complex, resp.; a total of 24.9 g. IV was obtained from fractions 1 and 2; fraction III gave Ph3B.NH3. The filtrates from fractions 1, 2, and 3 distd. with (CH2OH)2 and PhMe yielded 10.41 g. RB.O.CH2.CH2.O (IXa) (R = Ph, IX), b5 84-91.degree.; IX shaken with H2O gave PhB(OH)2, m. 215-16.degree.. IXa (R = Bu) (0.92 mole) treated with 1.84 g. PhMgBr and then with $\overline{NH3}$ gave 49% IV and 11% IX. IXa (R = BuO) (X) (0.146 mole) and 0.292 mole PhLi gave 41% IV and 13% IX. VIII.NH3 (2.13 g.) heated with 2 cc. V in 100 cc. PhMe, 10 cc. PhMe and its azeotropes distd. off, and the residue cooled gave 1.67 g. I, m. 187-8.degree.. VIII.NH3 (13.70 g.) in 100 cc. EtOH and 200 cc. H2O mixed with 6 cc. V in 6 cc. H2O yielded 10.97 g. I, m. 189-90.degree. (from EtOH and H2O). I (0.2321 g.) in 10 cc. MeOH and 1 cc. H2O mixed with 0.7491 g. HgCl2 and titrated with standard NaOH gave an equiv. wt. of 327; 0.7 g. PhHgCl, m. 252-3.degree., pptd. from the soln. I (5.33 g.) in MeOH and Me2CO acidified with HCl, the mixt. dild. with H2O, the borinic acid extd. with Et2O, and the ext. dried and distd. yielded 2.11 g. (Ph2B)2O, b1 210-13.degree., m. mostly at 104-5.degree.; it formed an oil with H2O. The Grignard deriv. from 7.29 g. Mg and 40.77 g. p-C6H4Br2 (consisting of 2 layers) added slowly with stirring to 18.38 g. X in 600 cc. Et2O at -60.degree., and the mixt. kept at room temp. overnight, hydrolyzed, esterified, and distd. gave 7.50 q. distillate, b2

144-72.degree., 2.87 g., b2 172-91.degree., 13.23 g., b2 191-5.degree., and 2.53 g., b2 195-7 degree; fractions 1 and 2 redistd. with (CH2OH)2 and PhMe gave 4.55 g. IXa (R = p-BrC6H4), b15 150-3.degree. m., 72-80.degree. [heated with H2O, it gave p-BrC6H4B(OH)2 (Xa), m. 254-6.degree.]; fraction 3 (11.38 g.) dissolved in N NaOH, the soln. extd. with Et2O, acidified with HCl, again extd. with Et2O, the ext. evapd., the residue dissolved in 500 cc. PhMe, the soln. treated with 5 cc. V, and the crude ppt. (6.95 g.) recrystd. gave VI, m. 236-7.degree.; the soln. from the acid titration (equiv. wt. 382) of VI extd. with Et2O gave (p-BrC6H4)2BOH, m. 82-4.degree. (from heptane). PhB(OH)2 (12.20 g.), 10.84 g. VII, and 800 cc. PhMe concd. by distn. to about 400 cc., the mixt. cooled, filtered, and the filter cake washed with dry Et2O gave 18.96 g. VII ester of PhB(OH)2, m. 212-14.degree.; a portion (7.12 g.) dissolved in 50 cc. hot EtOH and repptd. with ligroine gave 6.10 g. pure ester, m. 214-15.degree.. Similarly were prepd. the VII esters (% yield and m.p. given) of Xa, 58, 255-6.degree., and of p-MeOC6H4B(OH)2, 55, 216-18.degree.. VII (3 cc.) in 3 cc. H2O added to 4 q. 1-C10H7B(OH)2 in 10 cc. EtOH and 5 cc. H2O yielded 4.94 g. VII ester, m. 242-3.degree..

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L4 ANSWER 305 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN
AN 1956:16224 CAPLUS
DN 50:16224
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DN 50:16224 OREF 50:3353e-g

TI Alcoholysis of triarylboranes

AU Rondestvedt, Christian S., Jr.; Scribner, Richard M.; Wulfman, Carl E.

CS Univ. of Michigan, Ann Arbor

SO Journal of Organic Chemistry (1955), 20, 9-12 CODEN: JOCEAH; ISSN: 0022-3263

DT Journal

LA Unavailable

RN 2622-89-1 CAPLUS

CN Borinic acid, diphenyl- (6CI, 7CI, 8CI, 9CI) (CA INDEX NAME)

AB Refluxing 7.15 g. tri-1-naphthylborane (I) and 2.8 g. anhyd. H2NCH2CH2OH (II) 4.5 hrs. in 100 cc. anhyd. C6H6 gives 64% H2NCH2CH2 di-1-naphthylborinate, m. 205.degree. (decompn.). Refluxing triphenylborane (III) and II 12 hrs. in a N atm. gives 68% H2NCH2CH2 diphenylborinate, m. 188-90.degree. (decompn.). Refluxing 2 g. I 0.5 hr. with 100 cc. MeOH gives Me borate, C10H8, and unchanged I. Refluxing 10.8 g. III in 100 cc. MeOH 137 hrs. in a N atm. and distg. the residue of the evapd. mixt. give 5.5 cc. of an oil, b2.2 168-71.degree., m. 60-75.degree.; this, in petr. ether satd. with NH3, gives a compd., C18H18BNO, m. 152-3.degree., which seems to be Ph diphenylborinate-NH3. In a similar expt., 1 mole C6H6 is liberated with the formation of 34% triphenylboroxene, B303Ph3 (IV), m. 214-16.degree.. In another expt. when the residue, after removal of the MeOH, is treated in petr. ether with NH3, Me diphenylborinate-ammonia contaminated with IV or Me benzeneboronate is obtained. I and AcOH under various conditions give triacetoxyborane, C10H8; and I. I and HBr in C6H6 boiled 45 min. give 0.95 g. C10H8, 0.54 g. unchanged I, and 0.46 g. 1-naphthaleneboronic acid.

The cleavage is discussed in terms of electron-release in the transition state.

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ANSWER 306 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN
L4
     1956:16223 CAPLUS
AN
     50:16223
DN
     Studies in the naphthalene series. I. Synthesis of 3-amino-1-naphthoic
OREF 50:3353a-e
TΙ
     Vondracek, M.; Vecerek, B.
ΑU
     Karlova Univ., Prague
     Chemicke Listy pro Vedu a Prumysl (1955), 49, 772-5
CS
SO
     CODEN: CLPRAN; ISSN: 0366-6832
     Journal
DT
     Unavailable
ĿA
     2622-89-1, Borinic acid, diphenyl-
IT
         (esters)
      Borinic acid, diphenyl- (6CI, 7CI, 8CI, 9CI) (CA INDEX NAME)
RN
 CN
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Ph | | Ph— B— OH

A new synthesis has been worked out for 3-amino-1-naphthoic acid (I) giving an over-all yield of approx. 28%. Refluxing 30 min. a mixt. of 200 AΒ g. 1-naphthylamine, 140 ml. Ac20, and 100 ml. AcOH, adding portionwise 1100 ml. AcOH, cooling to 20.degree., treating the mixt. at 20.degree. with 220 g. Br in 400 ml. AcOH, stirring the white suspension of the Br deriv. 30 min., heating it up to 55.degree., and treating with 141 ml. HNO3 (d. 1.42) during 2 hrs. at 70.degree. gave 100 g. 1-bromo-3-nitro-4-acetonaphthalide (II), m. 228.degree.; another 100-g. portion of II was obtained from the mother liquor (total yield 50%). Heating 50 g. 4-bromo-1-acetonaphthalide, 18 g. CuCN, and 16 g. anhyd. C5H5N in the oil bath to 160.degree., and maintaining the reaction temp. by heating or cooling at 180-90.degree. 90 min., triturating the melt after cooling with 250 ml. concd. NH4OH, and crystg. the product from EtOH gave 29 g. 1-cyano-4-acetonaphthalide (III), m. 189.5.degree. (from EtOH). Nitration of 10 g. III with 4 ml. HNO3 (d. 1.5) while heating to 95.degree. yielded 10 g. 1-cyano-3-nitro-4-acetonaphthalide (IV), m. 270.5.degree. (from AcOH). Refluxing IV (10 g.) 3 hrs. with 140 ml. AcOH and 50 ml. HCl gave 85-90% 1-cyano-3-nitro-4-aminonaphthalene (V), m. 239.5.degree. (from AcOH). Adding a suspension of V in 200 ml. AcOH to a soln. of 8.2 g. NaNO2 in 55 ml. concd. H2SO4, stirring the mixt. below 20.degree. 30 min., treating the mixt. during 30 min. portionwise with 30 g. Cu2O, and pouring the mixt. after 45 min. into 1 1. H2O gave 69% 1-cyano-3-nitronaphthalene, m. 183.degree. (from EtOH) (VI). The same deriv. was obtained by heating 2 hrs. at 170.degree. a mixt. of 1 g. 1-bromo-3-nitronaphthalene, 0.4 g. CuCN, and 0.4 ml. anhyd. C5H5N; yield of VI 65-70%. Hydrolysis of 4 g. of VI by refluxing 3 hrs. with a mixt. of 50 ml. H2O, 50 ml. H2SO4, and 50 ml. AcOH gave 97% 3-nitro-1-naphthoic acid, m. 265.degree. (from EtOH). Hydrogenation over PtO2 in EtOH gave I, m. 179.degree. (from EtOH) (blue fluorescence); sulfate, sparingly sol. in H20.

L4 ANSWER 307 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN

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1955:46053 CAPLUS
AN
     49:46053
DN
OREF 49:8840d-f
    Aminoethyl diarylborinates; isolation of a stable unsymmetrical
TI
     organoboron compound
     Letsinger, Robert L.; Skoog, Ivan; Remes, Nathaniel
ΑU
     Northwestern Univ., Evanston, IL
CS
     Journal of the American Chemical Society (1954), 76, 4047-8
SO
     CODEN: JACSAT; ISSN: 0002-7863
     Journal
DT
     Unavailable
LΑ
     2622-89-1, Borinic acid, diphenyl-
IT
        (and esters)
     2622-89-1 CAPLUS
     Borinic acid, diphenyl- (6CI, 7CI, 8CI, 9CI) (CA INDEX NAME)
RN
CN
```

IT 62981-91-3, Borinic acid, di-1-naphthyl(prepn. of)
RN 62981-91-3 CAPLUS
CN Borinic acid, di-1-naphthalenyl- (9CI) (CA INDEX NAME)

Arylborinic acids can be isolated and characterized as their aminoethyl esters. The appropriate Grignard reagent (2 moles) treated with 1 mole of butylethylene borate or Bu borate and the product hydrolyzed with dil. HCl yielded the acids. Bu diphenylborinate was sepd. from Ph3B by distn. and from Bu benzeneboronate (I) by pptn. from Et2O with NH3. The NH3 complex, m. 64-7.degree., isolated in 48% yield, was converted in 80-90% yield to aminoethyl diphenylborinate, m. 189-90.degree. Pptn. from a PhMe soln. yielded 45% aminoethyl di-.alpha.-naphthylborinate, m. 192-3.5.degree.. Acid hydrolysis of these esters yielded diphenylborinic acid, an oil, and .alpha.-naphthylborinic acid, m. 105-6.degree.. C10H7MgBr (0.049 mole) and 0.05 mole I in Et2O at -60.degree. yielded 10.1 g. aminoethyl phenyl-.alpha.-naphthylborinate, m. 228-9.degree..

L4 ANSWER 308 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN AN 1952:58396 CAPLUS

DN 46:58396 OREF 46:9766e-h

TI Investigations of the insecticide action of organic compounds. VI AU Beran, F.; Prey, V.; Bohm, Helen

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Tech. Univ., Vienna
CS
    Mitt. Chem. Forsch.-Inst. Wirtsch. Osterr. (1952), 6, 54-6
SO
     Journal
DT
     Unavailable
LΑ
     73774-45-5, Borinic acid, bis(p-methoxyphenyl)-
IT
        (as insecticide)
     73774-45-5 CAPLUS
     Borinic acid, bis(4-methoxyphenyl) - (9CI) (CA INDEX NAME)
RN
CN
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cf. C.A. 46, 8802c. Org. B compds. were investigated for their action on Calandra granaria, Tenebrio molitor, Musca domestica, and Carausius morosus. While B compds. in which B is linked to C by O show no effect whatever, aromatic boric acids in which B is linked directly to C are excellent insecticides. No insecticides were found among different aliphatic compds. by the same method except among the malonic acid esters (loc. cit.). The following substances were tested, and their structural formulas, mol. wt., b.p., m.p., soly., and action by contact or vapor are tabulated: maleic acid-boric acid, boric acid-succinic acid anhydride, boric acid stearate, boric acid phthalate, boric acid dodecyl ester, phenylboric acid, m-chlorophenylboric acid, p-bromophenylboric acid, p-methoxyphenyl boric acid, bis(p-methoxyphenyl)boric acid, 1-naphthylboric acid, benzylboric acid, cyclohexylboric acid, trichloroethanol, 2,3,3-tribromobutyl alc., tris(hydroxymethyl)nitromethan e, 3,3,3 - trichloro - 1 - nitro - 2 - propanol, ClCH2CH(OH)CH2OH, chloral urethan, chlorosulfo hydrate, Ac2, acetylacetone, acetonylacetone, cyclopentanone, Cl3CCO2Et, malonic acid Et ester, (EtO2C)2CHBr, (EtO2C)2CBr2, di-Et trichloroethylidenemalonate, (MeO2C)2, oxaloacetic ester, (EtO2CCH2)2, levulinic acid, dichloroacetamide, thioacetamide, adipinic acid dinitrile, and 1,3-dichloro-2-chloromethyl-2-nitropropane.

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ANSWER 309 OF 309 CAPLUS COPYRIGHT 2003 ACS on STN
L4
     1939:35170 CAPLUS
AN
     33:35170
DN
OREF 33:4969i,4970a-e
    Organic boron compounds. III. Synthesis of aryl- and diarylboric acids
TI
     Mel'nikov, N. N.; Rokitskaya, M. S.
ΑU
     Zhurnal Obshchei Khimii (1938), 8, 1768-75
SO
     CODEN: ZOKHA4; ISSN: 0044-460X
     Journal
DT
     Unavailable
LΑ
     89566-59-6, Borinic acid, bis(p-chlorophenyl)-
TT
        (prepn. of)
     89566-59-6 CAPLUS
RN
     Borinic acid, bis(4-chlorophenyl)- (9CI) (CA INDEX NAME)
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CN

10085368.2

cf. C. A. 33, 4908.7. The method of Konig and Scharrnbeck (C. A. 25, 927) is used again in the prepn. of aryl- and diarylboric acids from the AB corresponding Mg compds. and iso-Bu borate in Et2O. All these compds. are cryst. white substances, fairly sol. in org. solvents and sparingly sol. in H2O. When stored over dehydrating agents, these acids form anhydrides. 2,5-MeClC6H3B(OH)2 (I) (from 4,2-ClBrC6H3Me), m. 184-6.degree... 4,2-MeBrC6H3B(OH)2 (from 3,4-Br2C6H3Me), m. 157.degree.. p-EtC6H4B(OH)2 (from p-EtC6H4Br), m. 108-11.degree. p-PhCH:CHB(OH)2 (from .omega.-bromostyrene), m. 138-41.degree. Biphenylboric acid (from bromobiphenyl), m. 185-90 degree.; as a by-product there is formed a little of dibiphenylboric acid, does not m. 300.degree.. 2,4,5-Me2BrC6H2B(OH)2 (from 4,6,1,3-Br2C6H2Me2), m. 206-11.degree.. 2,4,5-Me2ClC6H2B(OH)2 (from 4,6,1,3-BrClC6H2Me2), m. 155-7.degree.. 4,5-MeClC6H3B(OH)2 (from 2,4-ClBrC6H2Me), m. 242-7.degree.. (p-ClC6H4)2BOH, m. 75.degree., formed as a by-product in the prepn. of p-ClC6H4B(OH)2 (loc. cit.). (2,5-MeClC6H3)2BOH, m. 81.degree., is obtained as a by-product in the prepn. of I. p-ClC6H4CH2B(OH)2, (from p-ClC6H4CH2Br), m. 140 degree. The arylboric acids react with TlCl3 and TlBr3 to give Ar2TlX, which by interaction with Tl halides in H2O form ArTlX2 (X = Cl or Br) (cf. C. A. 30, 2182.9). The following new compds. were prepd. (2,4,5-Me2BrC6H2)2TlCl, decompg. 268.degree.. (2,4,5-Me2ClC6H2)2TlCl, decompg. 248.degree.. (4,5-MeClC6H3)2TlCl, decompg. 260.degree.. (2,5-MeClC6H3)2TlCl, m. 238.degree.. (4,2-MeBrC6H3)2TlCl, decompg. 223.degree.. (p-EtC6H4)2TlCl, decompg. 260.degree. (m-Me2C6H3)2TlBr, m. 196.degree. (2,4,5-Me2BrC6H2)2TlBr, decompg. 220.degree. (2,4,5-Me2ClC6H2)2TlBr, m. 190-5.degree. (4,5-MeClC6H3)2TlBr, decompg. 290.degree.. (4,2-MeBrC6H3)2TlBr, m. 253.degree. (2,5-MeClC6H3)2TlBr, m. 200.degree. (p-EtC6H4)2TlBr, decompg. 280.degree.. The above compds. were obtained in 60-95% yields, forming white cryst. substances. MeBrC6H3TlCl2, m. 174-7.degree., EtC6H4TlCl2, decompg. 155.degree., and .alpha.-Cl0H7TlCl2, m. 144.degree., are white cryst. compds. 2,4,5-Me2BrC6H2TlBr2, m. 192.degree., and 2,4,5-Me2ClC6H2TlBr2, m. 185-90.degree., are yellow cryst. compds. The following compds. form orange crystals. 4,5-MeClC6H3TlBr2, m. 185-8.degree. MeClC6H3TlBr2, m. 182.degree. MeBrC6H3TlBr2, m. 180.degree. p-EtC6H4TlBr2, m. 170.degree. Me2C6H3TlBr2, m. 215.degree.. .alpha.-C10H7TlBr2, m. 185.degree.. The above compds. were formed in 33-84% yields.

=> log y TOTAL SINCE FILE COST IN U.S. DOLLARS SESSION ENTRY 1616.79 1468.22 FULL ESTIMATED COST DISCOUNT AMOUNTS (FOR QUALIFYING ACCOUNTS) TOTAL SINCE FILE SESSION ENTRY -200.51 -200.51 CA SUBSCRIBER PRICE

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